

10/564,340

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NEWS	4	APR 28	EMBASE Controlled Term thesaurus enhanced
NEWS	5	APR 28	IMSRESEARCH reloaded with enhancements
NEWS	6	MAY 30	INPAFAMDB now available on STN for patent family searching
NEWS	7	MAY 30	DGENE, PCTGEN, and USGENE enhanced with new homology sequence search option
NEWS	8	JUN 06	EPFULL enhanced with 260,000 English abstracts
NEWS	9	JUN 06	KOREAPAT updated with 41,000 documents
NEWS	10	JUN 13	USPATFULL and USPAT2 updated with 11-character patent numbers for U.S. applications
NEWS	11	JUN 19	CAS REGISTRY includes selected substances from web-based collections
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NEWS	13	JUN 30	AEROSPACE enhanced with more than 1 million U.S. patent records
NEWS	14	JUN 30	EMBASE, EMBAL, and LEMBASE updated with additional options to display authors and affiliated organizations
NEWS	15	JUN 30	STN on the Web enhanced with new STN AnaVist Assistant and BLAST plug-in
NEWS	16	JUN 30	STN AnaVist enhanced with database content from EPFULL
NEWS	17	JUL 28	CA/CAPplus patent coverage enhanced
NEWS	18	JUL 28	EPFULL enhanced with additional legal status information from the epline Register
NEWS	19	JUL 28	IFICDB, IFIPAT, and IFIUDB reloaded with enhancements
NEWS	20	JUL 28	STN Viewer performance improved
NEWS	21	AUG 01	INPADOCDB and INPAFAMDB coverage enhanced
NEWS	22	AUG 13	CA/CAPplus enhanced with printed Chemical Abstracts page images from 1967-1998
NEWS	23	AUG 15	CAOLD to be discontinued on December 31, 2008
NEWS	24	AUG 15	CAPplus currency for Korean patents enhanced
NEWS	25	AUG 25	CA/CAPplus, CASREACT, and IFI and USPAT databases enhanced for more flexible patent number searching
NEWS	26	AUG 27	CAS definition of basic patents expanded to ensure comprehensive access to substance and sequence

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information

NEWS EXPRESS JUNE 27 08 CURRENT WINDOWS VERSION IS V8.3,  
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FILE 'HOME' ENTERED AT 15:37:52 ON 17 SEP 2008

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STRUCTURE FILE UPDATES: 16 SEP 2008 HIGHEST RN 1049663-83-3  
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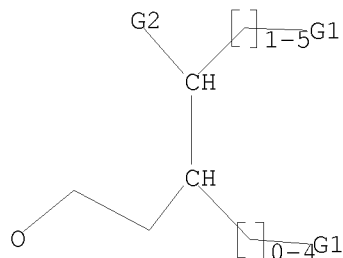
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L1 STRUCTURE UPLOADED

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=> dis  
L1 HAS NO ANSWERS  
L1 STR



G1 O, S, N

G2 CO<sub>2</sub>H, COOH, CN, CHO

Structure attributes must be viewed using STN Express query preparation.

=> s l1 sss full  
FULL SEARCH INITIATED 15:38:21 FILE 'REGISTRY'  
FULL SCREEN SEARCH COMPLETED - 6692517 TO ITERATE

14.9% PROCESSED 1000000 ITERATIONS 30 ANSWERS  
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)  
SEARCH TIME: 00.00.11

FULL FILE PROJECTIONS: ONLINE \*\*INCOMPLETE\*\*  
BATCH \*\*INCOMPLETE\*\*  
PROJECTED ITERATIONS: 6692517 TO 6692517  
PROJECTED ANSWERS: 158 TO 242

L2 30 SEA SSS FUL L1

=> fil hcap  
COST IN U.S. DOLLARS  
FULL ESTIMATED COST

	SINCE FILE	TOTAL
	ENTRY	SESSION
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FILE COVERS 1907 - 17 Sep 2008 VOL 149 ISS 12  
FILE LAST UPDATED: 16 Sep 2008 (20080916/ED)

HCAplus now includes complete International Patent Classification (IPC)  
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New CAS Information Use Policies, enter HELP USAGETERMS for details.

This file contains CAS Registry Numbers for easy and accurate  
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=> 12

L3 12 L2

=> d 12 ibib abs hitstr 1-12

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=> d 13 ibib abs hitstr 1-12

L3 ANSWER 1 OF 12 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2008:555891 HCAPLUS

DOCUMENT NUMBER: 148:518113

TITLE: Lactic acid polymers with high glass transition  
temperature

INVENTOR(S): Sodergard, Nils Dan Anders; Stolt, Erik Mikael;  
Inkinen, Saara

PATENT ASSIGNEE(S): Finland

SOURCE: U.S. Pat. Appl. Publ., 6pp.

CODEN: USXXCO

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 20080108759	A1	20080508	US 2007-936170	20071107
GB 2443625	A	20080514	GB 2006-22263	20061108
WO 2008056136	A1	20080515	WO 2007-GB4240	20071108
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW			
RW:	AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM			
PRIORITY APPLN. INFO.:	GB 2006-22263 A 20061108 US 2007-911557P P 20070413			

AB The invention relates to polymers comprising units derived from lactic acid, isosorbide, and a polycarboxylic acid having at least three carboxylic acid groups, the lactic acid units comprising at least 50% of the polymer. The incorporation of isosorbide and polycarboxylic acid units into lactic acid polymers can result in polymers having higher glass transition temperature. Preferably, 1,2,3,4-butanetetracarboxylic acid, 1,2,3,4,5,6-cyclohexanehexacarboxylic acid and/or their anhydrides are used as the polycarboxylic acid component.

IT 1022913-80-9P

RL: IMF (Industrial manufacture); PRP (Properties); PREP (Preparation)  
(lactic acid polymers with high glass transition temperature)

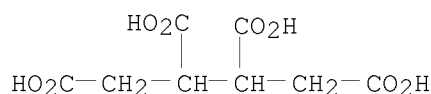
RN 1022913-80-9 HCAPLUS

CN D-Glucitol, 1,4:3,6-dianhydro-, polymer with 1,2,3,4-butanetetracarboxylic acid and (2S)-2-hydroxypropanoic acid (CA INDEX NAME)

CM 1

CRN 1703-58-8

CMF C8 H10 O8

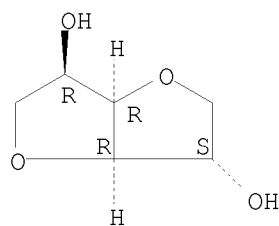


CM 2

CRN 652-67-5

CMF C6 H10 O4

Absolute stereochemistry. Rotation (+).

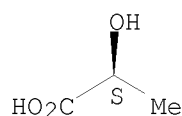


CM 3

CRN 79-33-4

CMF C3 H6 O3

Absolute stereochemistry. Rotation (+).



L3 ANSWER 2 OF 12 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2008:315746 HCAPLUS

DOCUMENT NUMBER: 148:333473

TITLE: Aqueous gas-barrier coatings and films coated therewith

INVENTOR(S): Hioki, Jun; Kuwata, Hideki; Okuzo, Kiyoshi; Yoshida, Mitsuo; Okamoto, Junji; Ozaki, Kunihiro; Kamoshita, Miyuki; Ueno, Reiko

PATENT ASSIGNEE(S): Unitika Ltd., Japan; Toyo Ink Mfg. Co., Ltd.

SOURCE: Jpn. Kokai Tokkyo Koho, 16pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2008056775	A	20080313	JP 2006-233731	20060830
PRIORITY APPLN. INFO.:			JP 2006-233731	20060830

AB Title coatings exhibiting high gas barrier property under high humidity comprise poly(vinyl alc.) (I), ethylene-maleic acid copolymer (II), at I/II weight ratio 97/3-10/90, and 0.1-70 parts to 100 parts I + II of low-mol.-weight polybasic carboxylic acids, and are applied on at least one side of thermoplastic resin films directly or via  $\geq 1$  undercoating layer to give the title films. Thus, an aqueous 10% solution of I (UF 040G) and

an aqueous 10% solution of II containing NaOH were blended at 55/45 and further blended with 60 parts/100 parts I + II of 1,2,3,4-butanetetracarboxylic acid to give a composition, which was applied on biaxially stretched nylon film (Emblem), dried, and heated to 200° for 15 s to give a gas-barrier film showing no discoloration and O permeability 17 mL/m<sup>2</sup>-day-MPa at 20° and relative humidity 85%.

IT 1010817-19-2P

RL: IMF (Industrial manufacture); PRP (Properties); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses)

(aqueous poly(vinyl alc.)/ethylene-maleic acid copolymer/polybasic carboxylic acid compns. as gas-barrier coatings for gas-barrier films)

RN 1010817-19-2 HCAPLUS

CN 1,2,3,4-Butanetetracarboxylic acid, polymer with (2Z)-2-butenedioic acid, ethene and ethenol, sodium salt (CA INDEX NAME)

CM 1

CRN 1010817-18-1

CMF (C8 H10 O8 . C4 H4 O4 . C2 H4 O . C2 H4)x

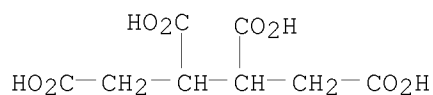
CCI PMS

CM 2

CRN 1703-58-8

CMF C8 H10 O8

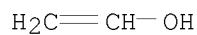
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CM 3

CRN 557-75-5

CMF C2 H4 O

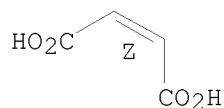


CM 4

CRN 110-16-7

CMF C4 H4 O4

Double bond geometry as shown.



CM 5

CRN 74-85-1

CMF C2 H4



L3 ANSWER 3 OF 12 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2008:267768 HCAPLUS

DOCUMENT NUMBER: 148:356534

TITLE: Lewis acid-containing fireproof copolymerized polyester materials and their manufacture

INVENTOR(S): Zheng, Jin; Tian, Xingyou; Cui, Ping; Li, Yong; Zhang, Xian; Yao, Xiayin

PATENT ASSIGNEE(S): Hefei Institutes of Physical Science, Chinese Academy of Sciences, Peop. Rep. China

SOURCE: Faming Zhuanli Shenqing Gongkai Shuomingshu, 14pp.

CODEN: CNXXEV

DOCUMENT TYPE: Patent

LANGUAGE: Chinese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CN 101130626	A	20080227	CN 2006-10086238	20060825

PRIORITY APPLN. INFO.: CN 2006-10086238 20060825

AB Title material comprise (a) copolymers from PET or PBT monomers and other copolymerizable monomers and (b) uniformly dispersed Lewis acids and are preferably prepared by mixing terephthalic acid (I) and ethylene or butylene glycol at 1:1.05-1.7 mol ratio, stirring with monomers, 0.01-30% (based on 100 parts final copolyesters) Lewis acids, and corresponding catalysts and aids at COOH/OH mol ratio of 1:1.05-1.7, esterifying at 250-260° and 2-3 kg/cm<sup>2</sup> for 2-3 h, removing excess H<sub>2</sub>O, further reacting at 265-275° and ≤700 Pa under N for 1.5 h, then at 265-275° and ≤70 Pa for 2-3 h. A copolyester was prepared at I/ethylene glycol at 1:1.05, glycerol at total COOH/OH of 1:1.05, and 30% blends of ZnCl<sub>2</sub>, AlCl<sub>3</sub>, GeCl<sub>4</sub>, Sn chloride, and Fe chloride in presence of Sb acetate and a matting agent and showed flexural strength 90.23 MPa and O index 33 without dripping.

IT 1012867-97-8P, 1,2,3,4-Butanetetracarboxylic acid-citric acid-ethylene glycol-glycerol-terephthalic acid copolymer  
 1012867-99-0P, 1,2,3,4-Butanetetracarboxylic acid-citric acid-ethylene glycol-glycerol-pentaerythritol-terephthalic acid copolymer  
 1012868-01-7P, 1,2,3,4-Butanetetracarboxylic acid-citric acid-ethylene glycol-glycerol-pentaerythritol-1,2,3-propanetricarboxylic acid-terephthalic acid copolymer  
 RL: IMF (Industrial manufacture); POF (Polymer in formulation); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses) (manufacture of Lewis acid-containing PET- or PBT-based copolyesters for fireproof materials)

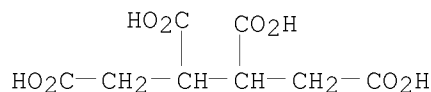
RN 1012867-97-8 HCAPLUS

CN 1,2,3,4-Butanetetracarboxylic acid, polymer with 1,4-benzenedicarboxylic acid, 1,2-ethanediol, 2-hydroxy-1,2,3-propanetricarboxylic acid and 1,2,3-propanetriol (CA INDEX NAME)

CM 1

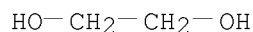
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CMF C8 H10 O8



CM 2

CRN 107-21-1  
 CMF C2 H6 O2

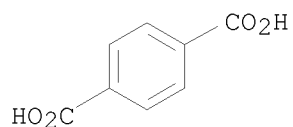


CM 3



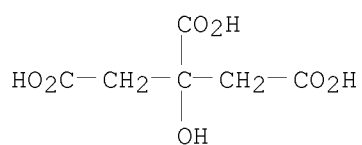
10/564,340

CRN 100-21-0  
CMF C8 H6 O4



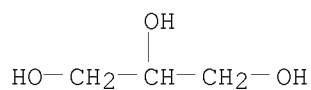
CM 4

CRN 77-92-9  
CMF C6 H8 O7



CM 5

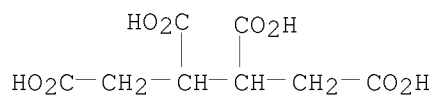
CRN 56-81-5  
CMF C3 H8 O3



RN 1012867-99-0 HCAPLUS  
CN 1,2,3,4-Butanetetracarboxylic acid, polymer with 1,4-benzenedicarboxylic acid, 2,2-bis(hydroxymethyl)-1,3-propanediol, 1,2-ethanediol, 2-hydroxy-1,2,3-propanetricarboxylic acid and 1,2,3-propanetriol (CA INDEX NAME)

CM 1

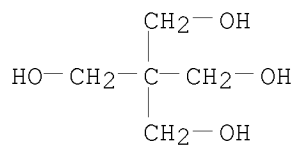
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CMF C8 H10 O8



CM 2

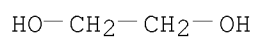
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CRN 115-77-5  
CMF C5 H12 O4



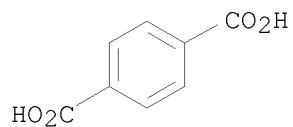
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CRN 107-21-1  
CMF C2 H6 O2



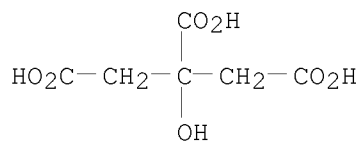
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CRN 100-21-0  
CMF C8 H6 O4



CM 5

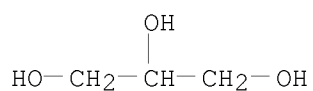
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CMF C6 H8 O7



CM 6

CRN 56-81-5  
CMF C3 H8 O3

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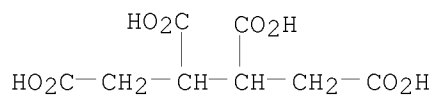
RN 1012868-01-7 HCAPLUS

CN 1,2,3,4-Butanetetracarboxylic acid, polymer with 1,4-benzenedicarboxylic acid, 2,2-bis(hydroxymethyl)-1,3-propanediol, 1,2-ethanediol, 2-hydroxy-1,2,3-propanetricarboxylic acid, 1,2,3-propanetricarboxylic acid and 1,2,3-propanetriol (CA INDEX NAME)

CM 1

CRN 1703-58-8

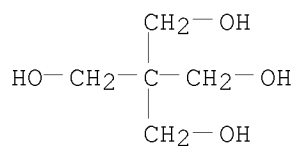
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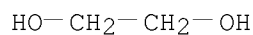
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CRN 107-21-1

CMF C2 H6 O2

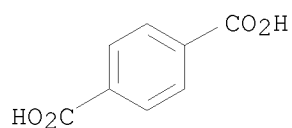


CM 4

CRN 100-21-0

CMF C8 H6 O4

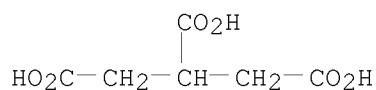
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CM 5

CRN 99-14-9

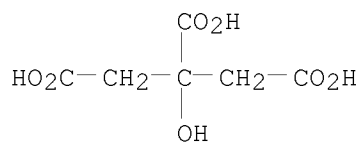
CMF C6 H8 O6



CM 6

CRN 77-92-9

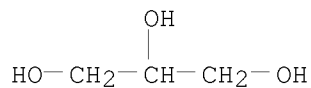
CMF C6 H8 O7



CM 7

CRN 56-81-5

CMF C3 H8 O3



L3 ANSWER 4 OF 12 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2008:26230 HCAPLUS

DOCUMENT NUMBER: 148:308551

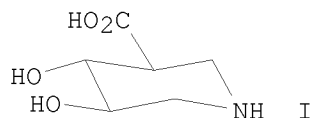
TITLE: A proline-catalyzed aldol approach to the synthesis of 1-N-iminosugars of the D-glucuronic acid type

AUTHOR(S): Chen, Chen; Yu, Biao

CORPORATE SOURCE: State Key Laboratory of Bio-organic and Natural Products Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai, 200032, Peop. Rep. China

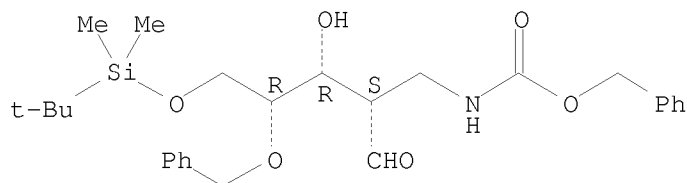
SOURCE: Tetrahedron Letters (2008), 49(4), 672-674

PUBLISHER: CODEN: TELEAY; ISSN: 0040-4039  
 Elsevier Ltd.  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 148:308551  
 GI



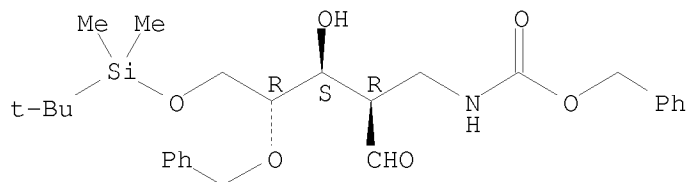
- AB A new synthetic route to 1-N-iminosugars of glucuronic acid type (e.g., I·HCl) has been developed employing proline-catalyzed aldol reaction as a key step.  
 IT 1009807-19-5P 1023304-41-7P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (asym. synthesis of iminosugars of D-glucuronic acid via proline-catalyzed stereoselective aldol addition of  $\beta$ -amino aldehyde and glyceric aldehyde, followed by heterocyclization)  
 RN 1009807-19-5 HCAPLUS  
 CN D-Lyxose, 2-deoxy-5-O-[(1,1-dimethylethyl)dimethylsilyl]-2-[[[(phenylmethoxy)carbonyl]amino]methyl]-4-O-(phenylmethyl)- (CA INDEX NAME)

Absolute stereochemistry.



- RN 1023304-41-7 HCAPLUS  
 CN D-Ribose, 2-deoxy-5-O-[(1,1-dimethylethyl)dimethylsilyl]-2-[[[(phenylmethoxy)carbonyl]amino]methyl]-4-O-(phenylmethyl)- (CA INDEX NAME)

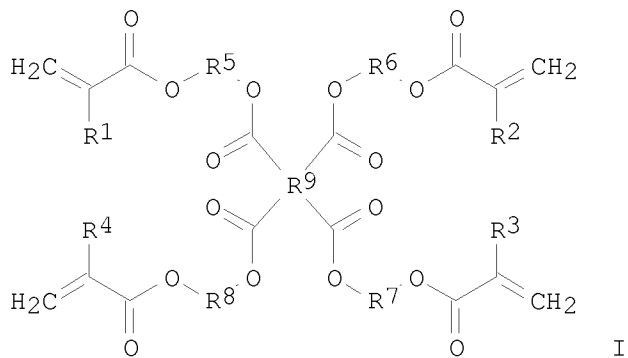
Absolute stereochemistry.



REFERENCE COUNT: 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 5 OF 12 HCAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 2007:1395285 HCAPLUS  
 DOCUMENT NUMBER: 148:34466  
 TITLE: Metal oxide composition for making cured film and laminate with good resistance to scratch  
 INVENTOR(S): Kaneda, Jun; Takahashi, Hayato; Shigemori, Kazunori; Hamada, Naohiro  
 PATENT ASSIGNEE(S): Toyo Ink Mfg. Co., Ltd., Japan  
 SOURCE: PCT Int. Appl., 49pp.  
 CODEN: PIXXD2  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2007138946	A1	20071206	WO 2007-JP60500	20070523
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW RW: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
PRIORITY APPLN. INFO.:			JP 2006-148854	A 20060529
			JP 2006-148855	A 20060529
OTHER SOURCE(S):		MARPAT 148:34466		
GI				



AB Disclosed is a metal oxide composition containing a dispersing agent (A) represented by the general formula I below and a metal oxide having an average primary particle diameter of 5-100 nm. In the formula I, R1-R4 independently represent a H atom or a Me group; R5-R8 independently represent an unsubstituted or substituted straight or branched chain alkylene group or alkylene oxyalkylene group; and R9 represents a tetravalent aromatic group or aliphatic group. Also disclosed are a cured film (e.g., coating) obtained by curing this metal oxide composition, and a laminate thereof useful for protection of instrument surface from scratching. Thus, heating biphenyltetracarboxylic dianhydride with Viscoat 300 (pentaerythritol acrylate) in cyclohexanone in the presence of 1,8-diazabicyclo[5.4.0]undec-7-ene, adding glycidyl methacrylate and heating gave a dispersant useful for dispersion of metal oxides..

IT 959611-38-2P 959611-39-3P 959611-40-6P  
959611-41-7P

RL: IMF (Industrial manufacture); NUU (Other use, unclassified); PREP (Preparation); USES (Uses)

(dispersant; manufacture of dispersants for film-forming compns. containing metal oxides for scratch prevention)

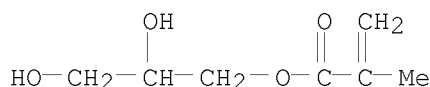
RN 959611-38-2 HCAPLUS

CN 1,2,3,4-Butanetetracarboxylic acid, 2-hydroxy-3-[(2-methyl-1-oxo-2-propen-1-yl)oxy]propyl ester, ester with 2,2-bis(hydroxymethyl)-1,3-propanediol 2-propenoate (CA INDEX NAME)

CM 1

CRN 5919-74-4

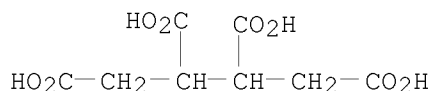
CMF C7 H12 O4



CM 2

CRN 1703-58-8

CMF C8 H10 O8



CM 3

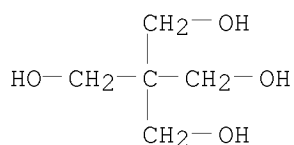
CRN 56093-53-9

CMF C5 H12 O4 . x C3 H4 O2

CM 4

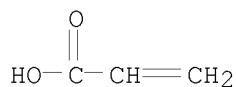
10/564,340

CRN 115-77-5  
CMF C5 H12 O4



CM 5

CRN 79-10-7  
CMF C3 H4 O2

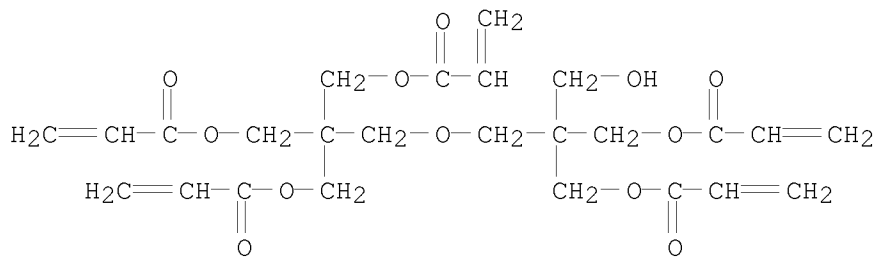


RN 959611-39-3 HCAPLUS

CN	1,2,3,4-Butanetetracarboxylic acid, 2-hydroxy-3-[(2-methyl-1-oxo-2-propen-1-yl)oxy]propyl 3-[3-[(1-oxo-2-propen-1-yl)oxy]-2,2-bis[[ (1-oxo-2-propen-1-yl)oxy]methyl]propoxy]-2,2-bis[[ (1-oxo-2-propen-1-yl)oxy]methyl]propyl 3-[(1-oxo-2-propen-1-yl)oxy]-2,2-bis[[ (1-oxo-2-propen-1-yl)oxy]methyl]propyl ester (CA INDEX NAME)
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CM 1

CRN 60506-81-2  
CMF C25 H32 O12

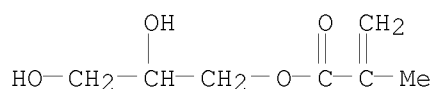


CM 2

CRN 5919-74-4  
CMF C7 H12 O4



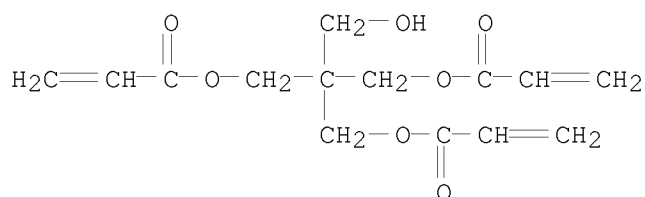
10/564,340



CM 3

CRN 3524-68-3

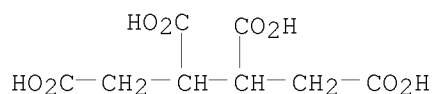
CMF C14 H18 O7



CM 4

CRN 1703-58-8

CMF C8 H10 O8



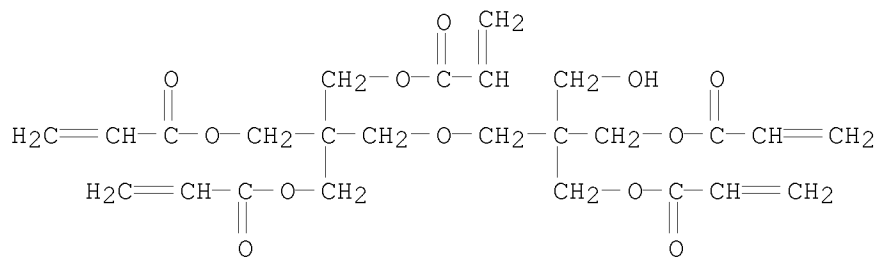
RN 959611-40-6 HCAPLUS

CN 1,2,3,4-Butanetetra-carboxylic acid, 2-hydroxy-3-[(2-methyl-1-oxo-2-propen-1-yl)oxy]propyl 3-[3-[(1-oxo-2-propen-1-yl)oxy]-2,2-bis[[ (1-oxo-2-propen-1-yl)oxy]methyl]propoxy]-2,2-bis[[ (1-oxo-2-propen-1-yl)oxy]methyl]propyl ester (CA INDEX NAME)

CM 1

CRN 60506-81-2

CMF C25 H32 O12

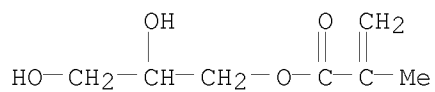


10/564,340

CM 2

CRN 5919-74-4

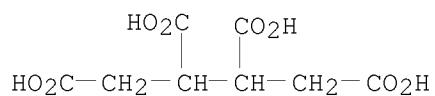
CMF C7 H12 O4



CM 3

CRN 1703-58-8

CMF C8 H10 O8



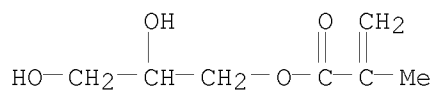
RN 959611-41-7 HCAPLUS

CN 1,2,3,4-Butanetetracarboxylic acid, 2-hydroxy-3-[(2-methyl-1-oxo-2-propen-1-yl)oxy]propyl 2-[(1-oxo-2-propen-1-yl)oxy]ethyl ester (CA INDEX NAME)

CM 1

CRN 5919-74-4

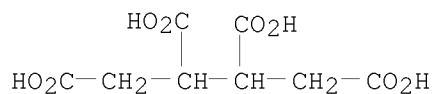
CMF C7 H12 O4



CM 2

CRN 1703-58-8

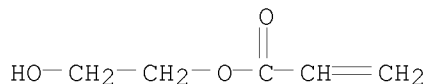
CMF C8 H10 O8



CM 3

CRN 818-61-1

CMF C5 H8 O3



REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 6 OF 12 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2007:1261316 HCAPLUS

DOCUMENT NUMBER: 148:102330

TITLE: Phase Behavior of Cetyltrimethylammonium Surfactants with Oligo Carboxylate Counterions Mixed with Water and Decanol: Attraction between Charged Planes or Spheres with Oligomeric Counterions

AUTHOR(S): Norrman, Jens; Piculell, Lennart

CORPORATE SOURCE: Division of Physical Chemistry 1, Center for Chemistry and Chemical Engineering, Lund University, Lund, SE-221 00, Swed.

SOURCE: Journal of Physical Chemistry B (2007), 111(47), 13364-13370

CODEN: JPCBFK; ISSN: 1520-6106

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Cetyltrimethylammonium surfactants with a range of oligo carboxylate anions bearing 2, 3, or 4 neg. charges have been synthesized, and their resp. behaviors in binary mixts. with water and in ternary mixts. with added decanol have been investigated. In binary mixts. with water, all surfactants formed nearly spherical micelles at high water contents; however, the interactions between micelles varied strongly with the number of charges in the counterion. Micelles with divalent counterions were generally miscible with water, whereas micelles with tri- or tetravalent counterions demixed in one concentrated and one dilute phase. Addition of decanol

resulted in all cases in the appearance of a lamellar phase, and all investigated oligo carboxylate anions (di-, tri-, and tetravalent) gave rise to a strong attraction between the lamellar planes, resulting in a limited swelling (up to 35-40 wt % water) of the lamellar phase in contact with excess water. These expts. confirm the theor. predicted influence of aggregate geometry (spheres or planes) on the attraction between colloidal aggregates neutralized by multivalent counterions. Further addition of decanol resulted in the appearance of a second birefringent phase in equilibrium with the lamellar phase. SWAXS showed this phase to be lamellar and to display short-range order that disappeared upon heating. This phase is identified as a lamellar gel phase (L $\beta$ -phase).

IT 1000407-41-9

RL: PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)

(aqueous phase behavior of cetyltrimethylammonium surfactants with oligo carboxylate counterions)

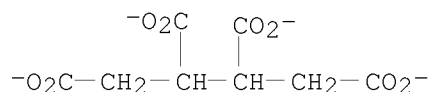
RN 1000407-41-9 HCAPLUS

CN 1-Hexadecanaminium, N,N,N-trimethyl-, 1,2,3,4-butanetetracarboxylate (4:1) (CA INDEX NAME)

10/564,340

CM 1

CRN 45170-94-3  
CMF C8 H6 O8



CM 2

CRN 6899-10-1  
CMF C19 H42 N

Me<sub>3</sub><sup>+</sup>N<sup>-</sup> (CH<sub>2</sub>)<sub>15</sub>-Me

REFERENCE COUNT: 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 7 OF 12 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2007:1110762 HCAPLUS

DOCUMENT NUMBER: 147:408089

TITLE: Heat- and oil-resistant hoses for automobiles

INVENTOR(S): Kano, Takamitsu; Tamura, Toru; Sugiura, Motoyuki

PATENT ASSIGNEE(S): NOF Corporation, Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 22pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2007254649	A	20071004	JP 2006-82776	20060324
PRIORITY APPLN. INFO.:			JP 2006-82776	20060324

AB Title hoses are formed from thermoplastic elastomer compns. prepared by dynamic crosslinking of material compns. of the following (A)-(D) with 0.05-5 parts/100 parts A of crosslinking agents: (A) 50-85 parts/100 parts (A + B) of acrylic rubber prepared by copolymn. of alkyl acrylates and/or alkoxyalkyl acrylates with 0.5-15% monomers having epoxy groups, (B) 15-50 parts/100 parts (A + B) of thermoplastic polyesters, (C) 1-35 parts/100 parts (A + B) of graft copolymers or their precursors consisting of olefin polymer segments formed from ethylene and polar monomers and vinyl copolymer segments formed from alkyl acrylate-containing vinyl monomers, where one segment forms dispersed phase in the other segment matrix, and (D) ≤60 parts/100 parts A of plasticizers. Thus, Et acrylate 250, Bu acrylate 230, and glycidyl methacrylate 20 g were polymerized in an aqueous emulsion, coagulated in aqueous CaCl<sub>2</sub>, and dried to give an acrylic rubber with Tg -27°, sep., 700 g ethylene-Et acrylate copolymer (NUC 6570)

2, was dispersed in an aqueous poly(vinyl alc.) solution, impregnated with Bz2O2

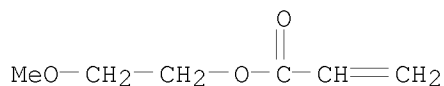
tert-Bu peroxy methacryloyloxyethyl carbonate 6, Bu acrylate 150, and 2-hydroxypropyl methacrylate 150 g, kept at 80-85° for 6 h, washed, and dried to give a grafting precursor. The acrylic rubber 65, Duranex 600JP 35, the precursor 5, ADK Cizer C 8 23, Irganox 1010 0.5, and Nonflex DCD 1 part were melt-kneaded, mixed with 0.2 part Rikacid BT-W, further kneaded, pelletized, and hot-pressed to give a sheet showing tensile strength 8.2 MPa initially and its change +26% after 1000 h at 150° or -45% after 1000 h in number 3 oil at 150°.

IT 951126-41-3P, Butyl acrylate-ethyl acrylate-glycidyl methacrylate-2-methoxyethyl acrylate-Rikacid BTW copolymer  
 RL: IMF (Industrial manufacture); POF (Polymer in formulation); PRP (Properties); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses)  
 (vulcanized rubber; heat- and oil-resistant hoses formed from acrylic rubber-based thermoplastic elastomer compns.)  
 RN 951126-41-3 HCAPLUS  
 CN 1,2,3,4-Butanetetracarboxylic acid, polymer with butyl 2-propenoate, ethyl 2-propenoate, 2-methoxyethyl 2-propenoate and 2-oxiranylmethyl 2-methyl-2-propenoate (CA INDEX NAME)

CM 1

CRN 3121-61-7

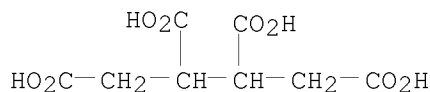
CMF C6 H10 O3



CM 2

CRN 1703-58-8

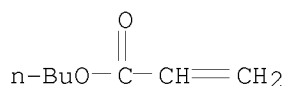
CMF C8 H10 O8



CM 3

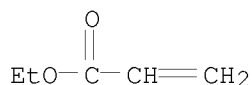
CRN 141-32-2

CMF C7 H12 O2



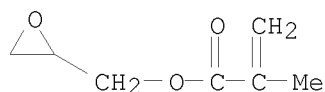
CM 4

CRN 140-88-5  
CMF C5 H8 O2



CM 5

CRN 106-91-2  
CMF C7 H10 O3



L3 ANSWER 8 OF 12 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2007:745450 HCAPLUS

DOCUMENT NUMBER: 147:189957

TITLE: Polyester composition containing hyperbranched polyamide

INVENTOR(S): Wang, Peng; Yang, Guisheng

PATENT ASSIGNEE(S): Shanghai Genius Advanced Material Co., Ltd., Peop. Rep. China

SOURCE: Faming Zhuanli Shenqing Gongkai Shuomingshu, 7pp.  
CODEN: CNXXEV

DOCUMENT TYPE: Patent

LANGUAGE: Chinese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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CN 1990545	A	20070704	CN 2005-10112394	20051230

PRIORITY APPLN. INFO.: CN 2005-10112394 20051230

AB The composition comprises poly(ethylene terephthalate) 40-80, high-branched polyamide 0.1-4, polymer-crystallization promoter 1-6, filler 10-50, and processing auxiliaries 0.5-1 parts. Thus, polyethylene terephthalate 80, high-branched polyamide (3,5-bis(4-aminophenoxy)benzoic acid homopolymer) 2, polypropylene glycol 4, processing auxiliary 1, and glass fibers 12 kg were kneaded and pelletized to give a title composition

IT 944548-29-2, 1,2-Diaminobenzene-1,2,3,4-Tetracarboxybutane copolymer

RL: POF (Polymer in formulation); PRP (Properties); USES (Uses)  
(fifth generation; polyester composition containing hyperbranched polyamide)

RN 944548-29-2 HCAPLUS

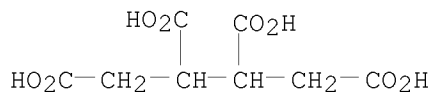
CN 1,2,3,4-Butanetetracarboxylic acid, polymer with 1,2-benzenediamine (CA

INDEX NAME)

CM 1

CRN 1703-58-8

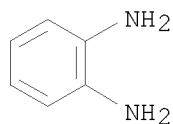
CMF C8 H10 O8



CM 2

CRN 95-54-5

CMF C6 H8 N2



L3 ANSWER 9 OF 12 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2007:666297 HCAPLUS

DOCUMENT NUMBER: 147:96985

TITLE: Synthetic fiber treatment agents preventing end breakage and fluff and good dyeability

INVENTOR(S): Sakakibara, Takuro; Kawamura, Koichi

PATENT ASSIGNEE(S): Takemoto Oil and Fat Co., Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 21pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2007154324	A	20070621	JP 2005-347402	20051201

PRIORITY APPLN. INFO.: JP 2005-347402 20051201

AB Title fiber treatment agents comprise (A) polyoxyalkylenes containing polyether compds. R1A1B1O2 72-99, polyethers (R3A2B2O)mY 0.5-27, and polyethers (R4B3A3O)nZ 0.1-10% ≥70, (B) nonionic surfactants polyoxyethylene C4-26 monoaliph. alkyl ether with Mn 180-1300, and (C) ≥1 ionic surfactants, wherein R1, R3 = H or monovalent C2-26 aliphatic acyl group; R2 = monovalent C1-26 aliphatic alkyl or C2-36 aliphatic alkenyl or aliphatic acyl group; R4 = H, monovalent C1-26 aliphatic alkyl or C2-26 aliphatic alkenyl or aliphatic acyl group; A1, A2, A3 = oxyethylene units with total ethylene oxide unit 2-200; B1 = random ≥2 C2-4 oxyalkylene units (total oxyalkylene unit = 2-200); B2, B3 = block, random, or block-random

$\geq 2$  C2-4 oxyalkylene units (total oxyalkylene unit = 2-200); Y = m-valent C2-26 or C4-26 aliphatic alkyl; Z = m-valent C4-26 fatty ester; and m, n = 2-4 integer. Thus, a polyester was melt-spun, solidified, treated with a 10% aqueous solution comprising an ethylene oxide-propylene oxide diblock

copolymer monobutyl ether 80, polyethylene glycol mono(3,5,5-trimethylhexyl) ether 18, sodium tetradecanesulfonate 1, and polyethylene glycol monodecyl ether 1 parts, stretched, wound, and twisted to give a test piece, showing no end breakage and fluff and good dyeability.

IT 942202-07-5 942202-13-3

RL: TEM (Technical or engineered material use); USES (Uses)  
(assumed monomer; synthetic fiber treatment agents comprising polyoxyalkylenes and ionic surfactants)

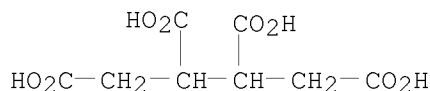
RN 942202-07-5 HCAPLUS

CN Oxirane, 2-methyl-, polymer with oxirane, 1,2,3,4-butanetetracarboxylate (4:1), diblock (CA INDEX NAME)

CM 1

CRN 1703-58-8

CMF C8 H10 O8



CM 2

CRN 697765-47-2

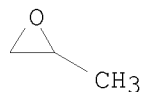
CMF (C3 H6 O . C2 H4 O)x

CCI PMS

CM 3

CRN 75-56-9

CMF C3 H6 O



CM 4

CRN 75-21-8

CMF C2 H4 O



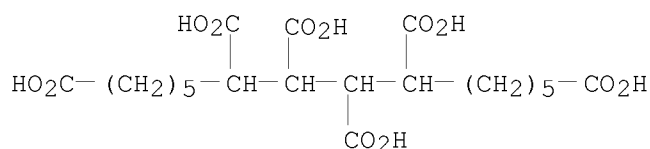


10/564,340

RN 942202-13-3 HCAPLUS  
CN Oxirane, 2-methyl-, polymer with oxirane, 1,6,7,8,9,14-tetradecanehexacarboxylate (6:1), diblock (CA INDEX NAME)

CM 1

CRN 78472-28-3  
CMF C20 H30 O12

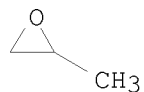


CM 2

CRN 697765-47-2  
CMF (C3 H6 O . C2 H4 O)x  
CCI PMS

CM 3

CRN 75-56-9  
CMF C3 H6 O



CM 4

CRN 75-21-8  
CMF C2 H4 O



L3 ANSWER 10 OF 12 HCAPLUS COPYRIGHT 2008 ACS on STN  
ACCESSION NUMBER: 2007:461150 HCAPLUS  
DOCUMENT NUMBER: 146:448449  
TITLE: Hydroxy oligocarboxylic esters effects on nerve and  
use for cutaneous and mucocutaneous organs  
INVENTOR(S): Yu, Ruey J.; Van Scott, Eugene J.  
PATENT ASSIGNEE(S): Yu, Ruey, J., USA; Van Scott, Eugene, J.  
SOURCE: PCT Int. Appl., 34 pp.  
CODEN: PIXXD2

DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2007047486	A2	20070426	WO 2006-US40179	20061013
WO 2007047486	A3	20080522		
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LV, LY, MA, MD, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW RW: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AP, EA, EP, OA US 20070093551 A1 20070426 US 2006-548945 20061012 AU 2006304295 A1 20070426 AU 2006-304295 20061013 CA 2625953 A1 20070426 CA 2006-2625953 20061013 EP 1937246 A2 20080702 EP 2006-825937 20061013 R: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LI, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR, AL, BA, HR, MK, RS PRIORITY APPLN. INFO.: US 2005-727419P P 20051017 US 2006-759525P P 20060117 US 2006-548945 A 20061012 WO 2006-US40179 W 20061013				

OTHER SOURCE(S): MARPAT 146:448449

AB A composition and method for producing a beneficial effect on a subject's nerve associated with at least one of a cosmetic condition, a dermatol. indication and a dental indication and another condition. The composition comprises a hydroxy- oligocarboxylic ester and is formulated for topical administration of the product to a subject to produce the beneficial effect. The method includes topically applying to the subject in a region where the beneficial effect is desired a hydroxy-oligocarboxylic ester in an amount effective to produce the beneficial effect. Tri-Me citrate 10 g was mixed with 90 g ointment prepared from white petrolatum 50, mineral oil 40, and white beeswax 10 parts. The composition thus prepared contained 10% tri-Me citrate in a water-non-washable ointment.

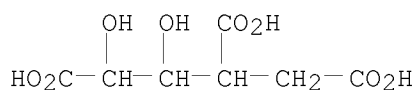
IT 934698-28-9D, esters 934698-30-3D, esters

RL: COS (Cosmetic use); THU (Therapeutic use); BIOL (Biological study);  
 USES (Uses)

(hydroxy oligocarboxylic esters effects on nerve and use for cutaneous and mucocutaneous organs)

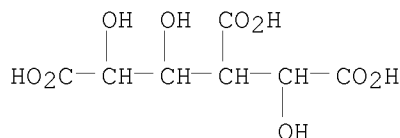
RN 934698-28-9 HCAPLUS

CN Hexaric acid, 3-carboxy-2,3-dideoxy- (CA INDEX NAME)



10/564,340

RN 934698-30-3 HCAPLUS  
CN Hexaric acid, 3-carboxy-3-deoxy- (CA INDEX NAME)



L3 ANSWER 11 OF 12 HCAPLUS COPYRIGHT 2008 ACS on STN  
ACCESSION NUMBER: 2007:223952 HCAPLUS  
DOCUMENT NUMBER: 146:298703  
TITLE: Porous organometallic framework materials prepared as  
calcined salts of polyvalent metals with  
polycarboxylic acids  
INVENTOR(S): Schubert, Markus; Mueller, Ulrich; Tonigold, Markus;  
Ruetz, Roger  
PATENT ASSIGNEE(S): Basf Aktiengesellschaft, Germany  
SOURCE: PCT Int. Appl., 43pp.  
CODEN: PIXXD2  
DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2007023134	A1	20070301	WO 2006-EP65442	20060818
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LV, LY, MA, MD, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW			
RW:	AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM			
DE 102005039623	A1	20070301	DE 2005-102005039623	20050822
CA 2620113	A1	20070301	CA 2006-2620113	20060818
EP 1922299	A1	20080521	EP 2006-792887	20060818
R:	AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LI, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR			
CN 101248034	A	20080820	CN 2006-80030753	20080222
KR 2008039985	A	20080507	KR 2008-706043	20080312
PRIORITY APPLN. INFO.:			DE 2005-102005039623A	20050822
			WO 2006-EP65442	W 20060818
AB	Porous organometallic framework materials (with a sp. surface area >10 m <sup>2</sup> /g), are prepared with a polyvalent metal ion coordinately bound with a polydentate ligand (i.e., with ≥2 coordinating sites) based on coordinating substituents on an organic compound that contains ≥2 atoms			

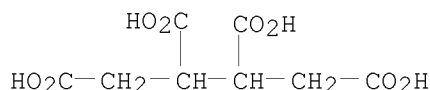
substituted by oxygen, sulfur, and nitrogen, in which the synthesis is carried out at a pressure >2 bars. The compds. are calcined prior to use. The invention also relates to the use of such porous organometallic framework materials. The organic compound is preferably a polydentate polycarboxylic acid, especially di-, tri-, or tetracarboxylic acids. Suitable metal ions include Be<sup>2+</sup>, Mg<sup>2+</sup>, Ca<sup>2+</sup>, Sr<sup>2+</sup>, Ba<sup>2+</sup>, Al<sup>3+</sup>, Ga<sup>3+</sup>, or In<sup>3+</sup>. The compns. are prepared in the presence of a nonaq. organic solvent, such as C1-6-alcs., DMSO, DMF, DEF, acetonitrile, toluene, dioxane, benzene, chlorobenzene, MEK, pyridine, THF, Et acetate, halogen-free C1-200-alkanes, sulfolane, glycol NMP,  $\gamma$ -butyrolactone, alicyclic alcs., and ketones and cyclic ketones. The compns. are useful as hydrogen and methane storage materials, and are polymerization catalysts for isobutene.

IT 927885-08-3P

RL: CAT (Catalyst use); PRP (Properties); SPN (Synthetic preparation); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses) (calcined; porous organometallic framework materials prepared as calcined salts of polyvalent metals with polycarboxylic acids)

RN 927885-08-3 HCAPLUS

CN 1,2,3,4-Butanetetracarboxylic acid, aluminum salt (3:4) (CA INDEX NAME)



● 4/3 A1

REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 12 OF 12 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2006:531129 HCAPLUS

DOCUMENT NUMBER: 146:275752

TITLE: Biodegradable network elastomeric polyesters from multifunctional aliphatic carboxylic acids and poly( $\epsilon$ -caprolactone) diols

AUTHOR(S): Nagata, Minoru; Kato, Keisuke; Sakai, Wataru; Tsutsumi, Naoto

CORPORATE SOURCE: Faculty of Human Environment, Kyoto Prefectural University, Shimogamo, Sakyo-ku, Kyoto, 606-8522, Japan

SOURCE: Macromolecular Bioscience (2006), 6(5), 333-339

CODEN: MBAIBU; ISSN: 1616-5187

PUBLISHER: Wiley-VCH Verlag GmbH & Co. KGaA

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Biodegradable elastomeric network polyesters were prepared from multifunctional aliphatic carboxylic acids such as tricarballic acid (Yt) or meso-1,2,3,4-butanetetracarboxylic acid (Xb) and poly( $\epsilon$ -caprolactone) (PCL) diols with mol. wts. of 530, 1 250 and 2 000 g·mol<sup>-1</sup>. Prepolymers prepared by a melt polycondensation were cast from DMF solution and postpolymd. at 280° for various periods of times to form a network. The resultant films were transparent, flexible and insol. in organic solvents. The network polyesters obtained were

characterized by IR absorption spectra, WAXS, d. measurement, DSC, and tensile test. YtPCL1250, and XbPCL1250 network polyester films showed good elastomeric properties with high ultimate elongation (540-590%), and low Young's modulus (2.5-3.3 MPa). The enzymic degradation was estimated by the weight loss of network films in a buffer solution with *Rhizopus deleamar* lipase at 37°. The degree and rate of degradation were significantly affected by the mol. weight of PCL diol, chemical structures of multifunctional aliphatic carboxylic acids and the morphol. of network films. The changes in the solid states of network films during the degradation were also estimated by the results of DSC and WAXS.

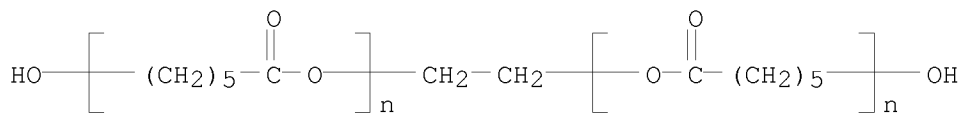
IT 927269-24-7P  
 RL: PRP (Properties); SPN (Synthetic preparation); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses)  
 (elastomer; biodegradable network elastomeric polyesters from multifunctional aliphatic carboxylic acids and polycaprolactone diols)

RN 927269-24-7 HCAPLUS

CN 1,2,3,4-Butanetetracarboxylic acid, (2R,3S)-rel-, polymer with  $\alpha, \alpha'$ -1,2-ethanediylbis[ $\omega$ -hydroxypoly[oxy(1-oxo-1,6-hexanediyl)]] (CA INDEX NAME)

CM 1

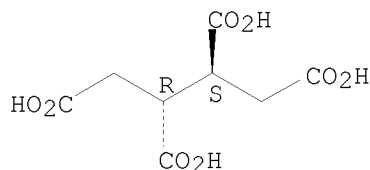
CRN 59692-54-5  
 CMF (C6 H10 O2)<sub>n</sub> (C6 H10 O2)<sub>n</sub> C2 H6 O2  
 CCI PMS



CM 2

CRN 4534-68-3  
 CMF C8 H10 O8

Relative stereochemistry.



REFERENCE COUNT: 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> fil reg

10/564,340

COST IN U.S. DOLLARS	SINCE FILE	TOTAL
	ENTRY	SESSION
FULL ESTIMATED COST	78.85	257.42
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE	TOTAL
	ENTRY	SESSION
CA SUBSCRIBER PRICE	-9.60	-9.60

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STRUCTURE FILE UPDATES: 16 SEP 2008 HIGHEST RN 1049663-83-3  
DICTIONARY FILE UPDATES: 16 SEP 2008 HIGHEST RN 1049663-83-3

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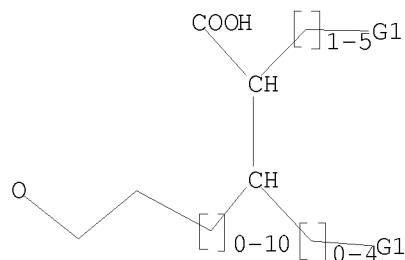
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L4 STRUCTURE UPLOADED

=> dis

L4 HAS NO ANSWERS

L4 STR



G1 O, S, N

G2 CO2H, COOH, CN, CHO

10/564,340

Structure attributes must be viewed using STN Express query preparation.

=> s l4 sss full  
FULL SEARCH INITIATED 15:44:16 FILE 'REGISTRY'  
FULL SCREEN SEARCH COMPLETED - 2166678 TO ITERATE

46.2% PROCESSED 1000000 ITERATIONS 180 ANSWERS  
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)  
SEARCH TIME: 00.00.08

FULL FILE PROJECTIONS: ONLINE \*\*INCOMPLETE\*\*  
BATCH \*\*INCOMPLETE\*\*  
PROJECTED ITERATIONS: 2166678 TO 2166678  
PROJECTED ANSWERS: 331 TO 449

L5 180 SEA SSS FUL L4

=> fil hcap		
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FULL ESTIMATED COST	180.20	437.62
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	ENTRY	SESSION
CA SUBSCRIBER PRICE	0.00	-9.60

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FILE COVERS 1907 - 17 Sep 2008 VOL 149 ISS 12  
FILE LAST UPDATED: 16 Sep 2008 (20080916/ED)

HCAplus now includes complete International Patent Classification (IPC) reclassification data for the second quarter of 2008.

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This file contains CAS Registry Numbers for easy and accurate substance identification.

=> l5  
L6 90 L5

10/564,340

=> 16 and (PD<20020711)  
22782685 PD<20020711  
(PD<20020711)  
L7 22 L6 AND (PD<20020711)

=> d 17 ibib abs hitstr 1-22

L7 ANSWER 1 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN  
ACCESSION NUMBER: 2004:203516 HCAPLUS  
DOCUMENT NUMBER: 140:237089  
TITLE: Composite fibrous substrates having carbohydrate  
sheaths and their manufacture  
INVENTOR(S): Offord, David A.; Ware, William; Millward, Dan B.;  
Soane, David S.; Young, Manfred A.  
PATENT ASSIGNEE(S): Nano-Tex, LLC, USA  
SOURCE: U.S. Pat. Appl. Publ., 12 pp., Cont.-in-part of U.S.  
Ser. No. 59,657.  
CODEN: USXXCO  
DOCUMENT TYPE: Patent  
LANGUAGE: English  
FAMILY ACC. NUM. COUNT: 7  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 20040048541	A1	20040311	US 2003-624095	20030721
US 6379753	B1	20020430	US 1999-274749	19990323 <--
WO 2002059404	A2	20020801	WO 2002-US2091	20020124
WO 2002059404	A3	20040219		
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW			
RW:	GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			
US 20020155771	A1	20021024	US 2002-59657	20020129
US 6617267	B2	20030909		
TW 240021	B	20050921	TW 2002-91104629	20020312
PRIORITY APPLN. INFO.:			US 1999-274749	A3 19990323
			US 2001-264181P	P 20010125
			US 2001-316358P	P 20010830
			WO 2002-US2091	A1 20020124
			US 2002-59657	A2 20020129
			US 1998-80185P	P 19980324
			US 1998-93820P	P 19980723
			US 1998-93911P	P 19980723
			US 1998-105890P	P 19981027
			US 1999-117641P	P 19990128
AB	The composite fibrous substrate comprises a core fiber and a carbohydrate sheath attached around the individual core fiber, wherein the carbohydrate sheath is adhered to itself by covalent bonds. The composite fibrous			



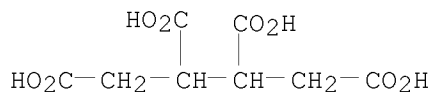
substrate is manufactured by treating a fibrous substrate containing core fibers (e.g., polyester fiber) with an aqueous solution of water-soluble carbohydrate (e.g., CM-cellulose sodium salt), a crosslinker (e.g., Freerez NFR), and optionally, a suitable crosslinker catalyst; heating the fibrous substrate to dryness; and curing at a temperature sufficient to cause reaction between the crosslinker and the carbohydrate. Individual synthetic fibers or yarns also can be applied, before weaving, knitting, stitch-bonding or other method of woven or non-woven substrate formation.

IT 667940-82-1  
 RL: TEM (Technical or engineered material use); USES (Uses)  
 (composite fibrous substrates having core fibers and carbohydrate sheaths)

RN 667940-82-1 HCAPLUS  
 CN Cellulose, carboxymethyl ether, sodium salt, polymer with  
 1,2,3,4-butanetetracarboxylic acid (9CI) (CA INDEX NAME)

CM 1

CRN 1703-58-8  
 CMF C8 H10 O8



CM 2

CRN 9004-32-4  
 CMF C2 H4 O3 . x Na . x Unspecified

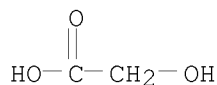
CM 3

CRN 9004-34-6  
 CMF Unspecified  
 CCI PMS, MAN

\*\*\* STRUCTURE DIAGRAM IS NOT AVAILABLE \*\*\*

CM 4

CRN 79-14-1  
 CMF C2 H4 O3



ACCESSION NUMBER: 2002:574364 HCAPLUS  
 DOCUMENT NUMBER: 138:123888  
 TITLE: Imidazole and imidazoline derivatives for powder coating application  
 AUTHOR(S): Toyota, Tsuyoshi  
 CORPORATE SOURCE: Shikoku Corporation, Japan  
 SOURCE: Proceedings of the Annual Meeting Technical Program of the FSCT (2000), 78th, 257-270  
 CODEN: PAMTCE; ISSN: 1536-9463  
 PUBLISHER: Federation of Societies for Coatings Technology  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English

AB Imidazole and imidazoline derivs. are widely used as a hardener for Epoxy Powder Coating and as an accelerator of Epoxy/Polyester Hybrid Powder Coating. We investigated the relation between reactivity, pot life, and chemical structure of the derivs. in Epoxy Powder Coating system. Some imidazole and imidazoline derivs. showed high reactivity as well as long pot life. Matting effect of the salts of imidazole/imidazoline with carboxylic acid was also investigated.

IT 490018-31-0P, 2-Phenylimidazole 1,2,3,4-butanetetracarboxylic acid salt  
 RL: CAT (Catalyst use); SPN (Synthetic preparation); PREP (Preparation); USES (Uses)  
 (imidazole and imidazoline derivs. as crosslinking promoters for epoxy resin powder coating application)

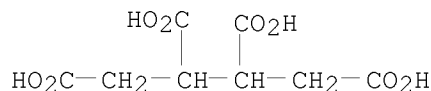
RN 490018-31-0 HCAPLUS

CN 1,2,3,4-Butanetetracarboxylic acid, compd. with 2-phenyl-1H-imidazole (1:?) (CA INDEX NAME)

CM 1

CRN 1703-58-8

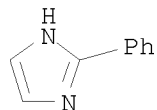
CMF C8 H10 O8



CM 2

CRN 670-96-2

CMF C9 H8 N2



L7 ANSWER 3 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1962:79614 HCAPLUS

DOCUMENT NUMBER: 56:79614  
 ORIGINAL REFERENCE NO.: 56:15606b-h  
 TITLE: Esters of amino acids and peptides  
 INVENTOR(S): Anderson, George Washington; Callahan, Francis M.  
 PATENT ASSIGNEE(S): American Cyanamid Co.  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 878732		19611004	GB 1959-29456	19590828 <--

AB Ger. 1118214. tert-Bu esters of amino acids and peptides are useful reagents for the synthesis of peptides. Ag carbobenzoxyglycinate (12.6 g.) in 100 cc. anhydrous ether, treated with 7.4 g. tert-BuI, the ether layer extracted with bicarbonate solution, and the extract worked up gave tert-Bu carbobenzoxyglycinate (I), b0.35 167°, n28D 1.4957, d2828 1.1065. I can be prepared also by adding to 20.9 g. carbobenzoxyglycine in iso-PrCOMe 2 g. p-toluenesulfonic acid and 23 g. isobutylene (II) at room temperature, keeping the mixture 18 hrs., diluting with 4% NaOH, and working up the extract. Similarly were prepared tert-Bu carbobenzoxy-L-tyrosinate, viscous oil, n26.5D 1.5222, d26.526.5 1.1212, tert-Bu carbobenzoxy-DL-valinate, n27D 1.4806, d2727 1.0329, tert-Bu carbobenzoxy-D-phenylalaninate, m. 78.5-80°. Carbobenzoxy-DL-phenylalanine (45 g.) in 1 cc. concentrated H2SO4 and 300 cc. iso-PrMeCO treated with 56.6 g. II, kept six days, diluted with 200 cc. 2% NaOH, and the extract worked up, gave tert-Bu carbobenzoxy-DL-phenylalaninate, viscous oil, n28D 1.5196, d2828 1.0914. Carbobenzoxyglycyl-L-phenylalanine (18.3 g.) in 500 cc. CH2Cl2 treated with 1 cc. H2SO4 and II with stirring, kept 20 hrs. at 25-30°, diluted with 200 cc. 5% KOH and the extract worked up gave tert-Bu carbobenzoxyglycyl-L-phenylalaninate, m. 61-3° (iso-Pr2O). I (15.3 g.) in 50 cc. absolute EtOH, hydrogenated in the presence of 10% Pd-C, treated with 4.75 g. H3PO3 gave tert-Bu glycinate phosphite (III), m. 155-7° (uncor.). III (10.65 g.) in 100 cc. ether shaken with 50 cc. 2N NaOH and the ether extract worked up gave tert-Bu glycinate, b2 30°, n24D 1.4227, d2424 0.9596. I hydrogenated as above, treated with butanetetra-carboxylic acid and filtered gave bis(tert-Bu glycinate) butanetetra-carboxylate, m. 148-50° (decomposition). Similarly were prepared tert-Bu L-tyrosinate, m. 142-3°, tert-Bu DL-phenylalaninate, b0.35 94-6°, n27D 1.4948, d2727 1.0135, tert-Bu D-phenylalaninate phosphite, m. 157-8°, tert-Bu L-leucinate phosphite, m. 163-4°, and tert-Bu glycyl-L-phenylalaninate, viscous oil, n25D 1.4987, d2525 1.0707. Phthaloylglycine (2.05 g.), 2.13 g. III, 1.40 cc. Et3N, and 17 cc. tetrahydrofuran treated with 2.27 g. dicyclohexylcarbodiimide (IV), the mixture refluxed 10 min., cooled to room temperature, enough AcOH added to decompose the unused IV, and the final filtrate diluted with 50 cc. H2O gave tert-Bu phthaloylglycinate (V), m. 165.0-5.5° (uncor.) (heptane-MeCOEt). V (740 mg.) treated with 9 cc. AcOH and 1.4 g. anhydrous HBr at room temperature and filtered after 2 min. gave 95% phthaloylglycylglycine, m. 236.5-7.0°. Anhydrous HCl (730 mg.) in 10 cc. di-Et phosphite, treated with 2.65 g. tert-Bu carbobenzoxyglycinate at 0°, the solution kept 2 mm. at 50°, cooled, treated with 20% NaOH, extracted with ether to remove unchanged ester, and the aqueous layer acidified gave 83.3% carbobenzoxyglycine, m.

119-20°. Carbobenzoxy-L-proline (1.99 g.), 20 cc. di-Et phosphite, 2.16 g. tert-Bu L-leucinate phosphite, 3 cc. tetra-Et pyrophosphite refluxed 0.5 hr. on steam bath, treated with ice-H<sub>2</sub>O mixture, and the precipitate washed with 10 cc. 5% KHC03 and filtered off gave tert-Bu carbobenzoxy-L-prolyl-L-leucinate, m. 89-90°, [ $\alpha$ ]<sub>25D</sub> -76.5° (5%, MeOH).

IT 859757-87-2P, Glycine, tert-butyl ester butanetetracarboxyl (2:1)  
 RL: PREP (Preparation)  
 (preparation of)

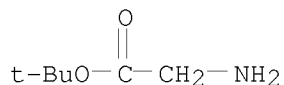
RN 859757-87-2 HCAPLUS

CN Glycine, tert-butyl ester butanetetracarboxyl (2:1) (7CI) (CA INDEX NAME)

CM 1

CRN 6456-74-2

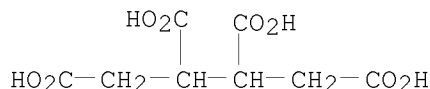
CMF C6 H13 N O2



CM 2

CRN 1703-58-8

CMF C8 H10 O8



L7 ANSWER 4 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1961:53911 HCAPLUS

DOCUMENT NUMBER: 55:53911

ORIGINAL REFERENCE NO.: 55:10310h-i,10311a-d

TITLE: Synthesis of a regular polyampholyte

AUTHOR(S): Marvel, C. S.; DeTommaso, G. L.

CORPORATE SOURCE: Univ. of Illinois, Urbana

SOURCE: Journal of Organic Chemistry (1960), 25, 2207-9

CODEN: JOCEAH; ISSN: 0022-3263

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB A regular polyampholyte, [SS(CH<sub>2</sub>)<sub>2</sub>CH(NH<sub>2</sub>)(CH<sub>2</sub>)<sub>2</sub>CH(CO<sub>2</sub>H)(CH<sub>2</sub>)<sub>22</sub>]<sub>n</sub>(I), was synthesized. 2,5-Diallyladipic acid (II) was distilled at 150-5°/0.05 mm., m. 102.5-3.5° (dilute alc.), in 89.9% yield. II (32.6 g.), a little Bz<sub>2</sub>O<sub>2</sub>, and 250 ml. C<sub>6</sub>H<sub>6</sub> treated dropwise near reflux with 53.3 g. thiolacetic acid, the mixture refluxed overnight, and the product recrystd. gave 39.7 g. 2,5-bis(3-mercaptopropyl)adipic acid diacetate (III), m. 148-9° (dilute alc.). Cyclohexane (200 mg.), 11.3 g. III, and 7.5 g.

AcCl refluxed 6 hrs. and the resulting viscous oil in CHCl<sub>3</sub> treated with gaseous NH<sub>3</sub> yielded 2,5-bis(3-mercaptopropyl)adipic acid, 2,5-bis(3-mercaptopropyl)adipamic acid, m. 145-6°, and 2,5-bis(3-mercaptopropyl)adipamide diacetate, m. 247° (HCONMe<sub>2</sub>). Distillation of the polymeric anhydride yielded a yellow oil, b<sub>0.15</sub> 181-6°, m. 66-7°, considered to be 2,5-bis(3-mercaptopropyl)cyclopentanone diacetate. 4-Carbethoxy-7-aminocarbethoxy-1,9-decadiene (IV) was prepared, b<sub>0.1</sub> 135-40°, n<sub>25D</sub> 1.4650. IV (56 g.), 1 l. xylene, and 4.8 g. NaH refluxed 6 hrs., poured into H<sub>2</sub>O, acidified, and extracted with C<sub>6</sub>H<sub>6</sub> gave 22.1 g. 3,6-diallyl-2-piperidone (V), b<sub>0.25</sub> 102-5°, m. 86.5-7.5° (dilute alc.). V (3 g.), 30 ml. cyclohexane, and some Bz<sub>2</sub>O<sub>2</sub> refluxed 4 hrs. with 10 ml. thiolacetic acid gave 4.5 g. 3,6-bis(3-mercaptopropyl)-2-piperidone diacetate (VI), m. 120-1° (dilute alc.). The hydrolysis of 3 g. VI was effected by stirring at room temperature 10 hrs. 2.4 g. KOH in 25 ml. 85% alc.; acidification gave a solid, which could not be purified. The polymeric Pb mercaptide was prepared from Pb(OAc)<sub>2</sub> and the dithiol in alc.; the dithiol regenerated by bubbling in H<sub>2</sub>S, filtering, and evaporating gave 2 g. 3,6-bis(3-mercaptopropyl)-2-piperidone (VII), m. 83.5-5.0° (cyclohexane). VII (2 g.), 2 g. KOH, 1.5 g. lauric acid, 25 ml. distilled H<sub>2</sub>O, 15 mg. selenous acid, and 1 drop Antifoam A were used in all polymerizations. After a 7 day polymerization period, the polymer latices were coagulated by pouring into 600 ml. 50% aqueous Me-OH containing 5 ml.

concentrated

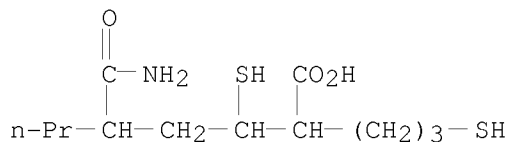
HCl to give a yellow or pink solid. The polymer was purified by dissoln. in hot (CHCl<sub>3</sub>)<sub>2</sub> and reprecipn. with 80% aqueous MeOH, m. 212-59°. The polymer (0.5 g.) was refluxed 24 hrs. with 25 ml. concentrated HCl, the residue filtered off, and dried; it showed infrared maximum which indicated hydrolysis of several isolated lactam functions. The acid filtrate evaporated and the residue lyophilized in H<sub>2</sub>O gave 83% I, white rubbery polymer, m. 247-64°. Neutralization of the amino-HCl with KOH gave a white rubbery solid, insol. in hot H<sub>2</sub>O and in wet and dry tetrahydrofuran and dioxane. The infrared spectra were given for the compds.

IT 857221-98-8P, Octanoic acid, 5-carbamoyl-3-mercapto-2-(3-mercaptopropyl)-

RL: PREP (Preparation)  
(preparation of)

RN 857221-98-8 HCAPLUS

CN Octanoic acid, 5-(aminocarbonyl)-3-mercapto-2-(3-mercaptopropyl)- (CA INDEX NAME)



L7 ANSWER 5 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1958:82753 HCAPLUS

DOCUMENT NUMBER: 52:82753

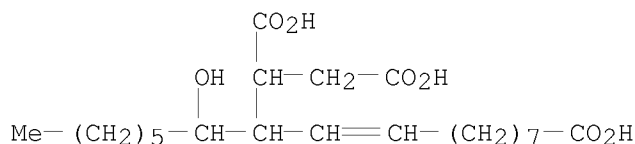
ORIGINAL REFERENCE NO.: 52:14664a-b

TITLE: Metal salt of maleic acid half ester

INVENTOR(S): Onishi, Hizoko

PATENT ASSIGNEE(S): Yoshitomi Pharmaceutical Industries, Ltd.  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	JP 32008176	B4	19570925	JP	<--
GI	For diagram(s), see printed CA Issue.				
AB	Me ricinoleate (31 g.) and 10 g. (: CHCO)20 heated 6-8 hrs. at 100-10° and the product extracted with 45 ml. C6H6 and concentrated gave 39 g. Me(CH2)5CH.CH[CH:CH(CH2)7CO2Me].CH(CH2CO2H).CO.O (I). I (41 g.) at 40-50° neutralized with 10% NaOH, then treated with BaCl2 (1:5), and the product extracted with C6H6 gave 45 g. Ba salt of I, yellow brown oil, which is useful as a stabilizing agent for synthetic resins.				
IT	860383-73-9P, 4-Dodecene-1,2,12-tricarboxylic acid, 3-(1-hydroxyheptyl)-, Ba salt RL: PREP (Preparation) (preparation of)				
RN	860383-73-9 HCAPLUS				
CN	4-Dodecene-1,2,12-tricarboxylic acid, 3-(1-hydroxyheptyl)-, barium salt (1:1) (CA INDEX NAME)				



● Ba

L7 ANSWER 6 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1955:84193 HCAPLUS  
 DOCUMENT NUMBER: 49:84193  
 ORIGINAL REFERENCE NO.: 49:15856d-i,15857a-e  
 TITLE: The synthesis of furan, thiophene, and pyrrole-3,4-dicarboxylic esters  
 AUTHOR(S): Kornfeld, E. C.; Jones, R. G.  
 CORPORATE SOURCE: Lilly Research Labs., Indianapolis, IN  
 SOURCE: Journal of Organic Chemistry (1954), 19, 1671-80  
 CODEN: JOCEAH; ISSN: 0022-3263  
 DOCUMENT TYPE: Journal  
 LANGUAGE: Unavailable  
 OTHER SOURCE(S): CASREACT 49:84193  
 GI For diagram(s), see printed CA Issue.  
 AB R.CR':C(CO2R'').C(CO2R''):CH (I) (R = O, S, or NH) are prepared by cyclization of appropriate 1,4-dicarbonyl derivs. Formylation of di-Et succinate in the presence of Na gives 65% di-Et α-formylsuccinate which (252.5 g.) is condensed with 210 g. HC(OEt)3 in 75 cc. EtOH and 3

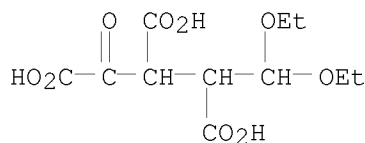
drops concentrated H<sub>2</sub>SO<sub>4</sub> (or HCl) 17 h. on a steam bath and the mixture fractionated, giving 82% (or 59%) R'O<sub>2</sub>CCH<sub>2</sub>CH[CH(OR'')<sub>2</sub>]CO<sub>2</sub>R' (II) (R' = R'' = Et, IIa), b<sub>7</sub> 135-45°, n<sub>24D</sub> 1.4283, d<sub>25</sub> 1.044. Adding dropwise (70 min.) a mixture of 400 g. IIa and 214 g. HCO<sub>2</sub>Et to 40 g. powdered Na in 1 l. Et<sub>2</sub>O and 15 cc. EtOH, stirring the mixture 1.5 h., keeping it overnight, and fractionally distilling the residue of the washed ether solution gives 87% R'O<sub>2</sub>CCH(CHO)CH[CH(OR')<sub>2</sub>]CO<sub>2</sub>R' (III) (R' = Et, IIIa), b<sub>0.8</sub> 124°, n<sub>25D</sub> 1.4682, d<sub>25</sub> 1.117. II (R' = R'' = Me), prepared in 62% yield, b<sub>6</sub> 125-30°, n<sub>25D</sub> 1.4315, when formylated gives 48% (or 75% based on recovered II) III (R' = Me) (IIIb), b<sub>0.5</sub> 125-30°, n<sub>25D</sub> 1.4752. Adding (5 min.) 165 g. IIIa to 330 cc. concentrated H<sub>2</sub>SO<sub>4</sub> at 48-52° with stirring, keeping the mixture 5 min. at 50°, pouring it at 0° onto ice, and distilling the residue of the Et<sub>2</sub>O layer gives 68% I (R = O, R' = H, R'' = Et), b<sub>6</sub> 125-7°, n<sub>25D</sub> 1.860, d<sub>2</sub>, 1.165. Other condensing agents used are [temperature, time (min.), and yield given]: POCl<sub>3</sub>, 100°, 30, 30%; HF, 25°, 60, 40%; ZnCl<sub>2</sub> in C<sub>6</sub>H<sub>6</sub>, 80°, 60, 43%; H<sub>3</sub>PO<sub>4</sub>, 55-85°, 10, 21%; BF<sub>3</sub> in C<sub>6</sub>H<sub>6</sub>, 30°, 120, 14%. Cyclization of IIIb with H<sub>2</sub>SO<sub>4</sub> gives 66% I (R = O, R' = H, R'' = Me), m. 49-50°. Heating 155 g. IIIa 2 h. in 650 cc. PhMe and 110 g. P<sub>2</sub>S<sub>5</sub> and fractionally distilling the PhMe layer gave 60 g. of a fraction, b<sub>5</sub> 100-60°, which is heated with 20 g. NaOH in 50 cc. H<sub>2</sub>O and 50 cc. EtOH and the residue of the evaporated solution acidified with concentrated HCl, giving 35% I (R = S, R' = R'' = H), m. 225-6° (di-Me ester, 87%, m. 60-1°; di-Et ester, 87%, b<sub>8</sub> 156-7°). Evaporating in vacuo a solution of 55.5 g. IIIa in 75 cc. ether and 4 g. NH<sub>3</sub> in 25 cc. EtOH, adding the residual sirup to 110 cc. concentrated H<sub>2</sub>SO<sub>4</sub> with stirring at 45°, keeping the mixture 5 min., and pouring it into H<sub>2</sub>O gave 49% I (R = NH, R' = H, R'' = Et) (IV), m. 153-5°, also obtained in 9% yield by heating IIIa with a mixture of NH<sub>4</sub>OAc, NH<sub>4</sub>Cl, and AcOH. Refluxing 1.5 g. IV with 3 g. NaOH in 40 cc. H<sub>2</sub>O-EtOH (1:1) gives 55 % free acid, m. 290-2° (decomposition). I (R = NMe, R' = H, R'' = Et), 45.5%, b<sub>0.5</sub> 163-7°, m. 47.5-8°; free acid, plates, m. 275-6° (decomposition). I (R = NPh, R' = H, R'' = Et), 49%, b<sub>1</sub> 210-15°, b<sub>0.5</sub> 200°, m. 48°; free acid, m. 278-80° (decomposition). Treating 90 g. di-Et acetylsuccinate, 74 g. HC(OEt)<sub>3</sub>, and 23 cc. absolute EtOH 64 h. with 10 drops concentrated H<sub>2</sub>SO<sub>4</sub>, steam distilling the mixture, adding 2 cc. N(CH<sub>2</sub>CH<sub>2</sub>OH)<sub>3</sub>, and distilling it in vacuo give 90% di-Et acetylsuccinate di-Et ketal (V), b<sub>0.1</sub>, 95°, n<sub>25D</sub> 1.4405, d<sub>25</sub> 1.041, which (82 g.), treated with 30 g. HCO<sub>2</sub>Et and 8 g. Na in 300 cc. ether, gives 50% di-Et 2-formyl-3-α-diethoxyethylsuccinate (VI) and 38% unchanged V. Cyclization of 45 g. VI with H<sub>2</sub>SO<sub>4</sub> gives 57% I (R = O, R' = Me, R'' = Et) (VII), b<sub>9</sub> 133-6°, b<sub>6</sub> 127-9°, n<sub>25D</sub> 1.4683, d<sub>25</sub> 1.139 (free acid, m. 236-7°). Treating 79 g. VI in 200 cc. ether with 45 g. 19% NH<sub>3</sub> in absolute EtOH, evaporating the mixture in vacuo, warming the residue with 175 cc. concentrated H<sub>2</sub>SO<sub>4</sub> at below 55°, pouring the cooled mixture after 5 min. onto 700 g. ice, and extracting it with AcOEt gave 21% I (R = NH, R' = Me, R'' = Et), m. 124-5°, and, from the mother liquor, 30% VII, b<sub>6</sub> 123-6°, n<sub>25D</sub> 1.4677. Condensation of 0.5 mol VI and 0.5 mol (CO<sub>2</sub>Et)<sub>2</sub> in the presence of 0.56 g. Na in 250 cc. ether gives 11.5% unchanged VI and 75% di-Et 2-ethoxalyl-3-diethoxymethylsuccinate (VIII) which could not be distilled and which (100 g.) distilled in vacuo decompose, giving 91% di-Et 2-carbethoxy-3-ethoxymethylenesuccinate, b<sub>1</sub>, 162-5°, b<sub>8</sub> 186-90°, n<sub>25D</sub> 1.4618, d<sub>25</sub> 1.112. Adding 140 g. crude VIII to 300 cc. concentrated H<sub>2</sub>SO<sub>4</sub>, warming the mixture 10 min. at

50°, pouring it at 10° onto 1.5 kg. ice, extracting with Et<sub>2</sub>O, washing the ether solution with 400 cc. saturated NaHCO<sub>2</sub> solution, and distilling the residue of the Et<sub>2</sub>O extract gave 12% tri-Et 2,3,4-furantricarboxylate, b<sub>0.4</sub> 152-3° (tri-Me ester, m. 108-9°). Acidification of the NaHCO<sub>3</sub> solution and extraction with Et<sub>2</sub>O gives 84% 3-hydroxy-4,5-dicarbethoxy-2-pyrone (IX), needles, m. 93-4°. Adding (0.5 h.) 49 g. (CO<sub>2</sub>CMe<sub>2</sub>)<sub>2</sub> and 67 g. IIa in 150 cc. absolute Et<sub>2</sub>O to 6 g. powdered Na in 150 cc. Et<sub>2</sub>O, stirring the mixture 2 h., keeping it overnight, adding ice, acidifying the aqueous layer with H<sub>2</sub>SO<sub>4</sub>, and extracting with Et<sub>2</sub>O gives 58% di-Et 2-diethoxymethyl-3-tert-butoxalylsuccinate, which cyclized with concentrated H<sub>2</sub>SO<sub>4</sub> gives 81% IX; it gives a pos. FeCl<sub>3</sub> test and does not react with SOCl<sub>2</sub>. Adding (0.5 h.) 510 g. CH<sub>2</sub>BrCOCO<sub>2</sub>Et to 560 g. NaO<sub>2</sub>CCOCH<sub>2</sub>CO<sub>2</sub>Et in 1 l. Et<sub>2</sub>O, keeping the mixture overnight, adding 1.5 l. H<sub>2</sub>O, and evaporating the Et<sub>2</sub>O layer gives 70% 2,3,4-tricarbethoxy-3-hydroxy-3,4-dihydrofuran, m. 79°, which treated with 1 l. concentrated H<sub>2</sub>SO<sub>4</sub> 10 min. at about 50° gives 80% tri-Et 2,3,4-furantricarboxylate, b<sub>3</sub> 175-80°, m. 45° (tri-Me ester m. 108-9°).

IT 850645-19-1, Tricarballic acid, α-(diethoxymethyl)-γ-oxo- (esters)

RN 850645-19-1 HCAPLUS

CN 1,2,3-Butanetricarboxylic acid, 4,4-diethoxy-1-oxo- (CA INDEX NAME)



L7 ANSWER 7 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1955:69135 HCAPLUS

DOCUMENT NUMBER: 49:69135

ORIGINAL REFERENCE NO.: 49:13267i,13268a-c

TITLE: Synthesis of 3-carboxyl-4-carboxymethylpimelic acid

AUTHOR(S): Pino, Piero

CORPORATE SOURCE: Politec., Milan

SOURCE: Atti accad. nazl. Lincei, Rend., Classe sci. fis., mat. e nat. (1954), 16, 640-5

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB The purpose was to demonstrate the identity of 3-carboxyl-4-(carboxymethyl)pimelic acid (I) with the hexanetetracarboxylic acid (II) obtained by the chromic oxidation of cevine and germin. The infrared spectrum of I obtained synthetically showed it is different from II (cf. Elming, et al., C.A. 49, 1068h). The condensation of Et 2-carboxy-3-(carboxymethyl)adipate with ClCH<sub>2</sub>CO<sub>2</sub>Et as a step in the synthesis of I did not take place. Condensation of 1,4-carboethoxy-1-butene (III) with EtO<sub>2</sub>CCH<sub>2</sub>CH(CO<sub>2</sub>Et)<sub>2</sub> (IV) gave the Et ester of HO<sub>2</sub>CCH<sub>2</sub>C(CO<sub>2</sub>H)<sub>2</sub>CH(CH<sub>2</sub>CO<sub>2</sub>H)CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H (V), saponified and decarboxylated to I. Na (0.23 g.) dissolved in 10 ml. absolute alc. was treated with 3 g. IV and 1.7 g. III (cf. Farmer and Hughes, C.A. 29, 1386.9), the mixture allowed to



stand 3 days, the alc. evaporated in vacuo, H<sub>2</sub>O added, the oil which separated extracted with Et<sub>2</sub>O, the extract dried with MgSO<sub>4</sub>, the Et<sub>2</sub>O evaporated, and the residue distilled, yielding 1.2 g., Et ester of V, b. 193°/0.4-0.5 mm., n<sub>20</sub>D 1.4559. V (0.5 g.) heated 8 hrs. at 180° with 10 ml. of 0.5N HCl in a sealed tube, the mixture extracted continuously 4 days with Et<sub>2</sub>O, the extract dried with MgSO<sub>4</sub>, the Et<sub>2</sub>O evaporated, and the residue

crystallized from

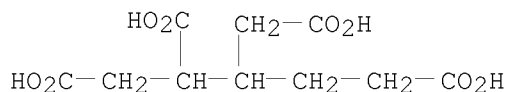
Et<sub>2</sub>O-Me<sub>2</sub>CO yielded 100 mg. I, m. 159-60°, mixed. m.p. with II 148-50°. Tetra-Me ester (VI) of I was prepared by treating 70 mg. I in Et<sub>2</sub>O with an excess of CH<sub>2</sub>N<sub>2</sub> in Et<sub>2</sub>O, and distilling in vacuo, b<sub>0.2</sub> 150-5°, n<sub>20</sub>D 1.459. The infrared spectra of VI and of the Et ester of II were taken in CS<sub>2</sub> and CCl<sub>4</sub> solns.

IT 875226-88-3P, 1,2,5-Pentanetricarboxylic acid, 3-(carboxymethyl)-  
RL: PREP (Preparation)

(preparation of)

RN 875226-88-3 HCAPLUS

CN 1,2,5-Pentanetricarboxylic acid, 3-(carboxymethyl)- (CA INDEX NAME)



L7 ANSWER 8 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1955:28006 HCAPLUS

DOCUMENT NUMBER: 49:28006

ORIGINAL REFERENCE NO.: 49:5394d-i, 5395a-i, 5396a-e

TITLE: Diene syntheses. XLII. Diene syntheses with unsymmetrical addends. Preparation of certain 1,2-substituted dienes and their applicability in diene syntheses

AUTHOR(S): Alder, Kurt; Haydn, Joseph; Heimbach, Karl; Neufang, Karl; Hansen, Gisela; Gerhard, Walter

CORPORATE SOURCE: Univ. Cologne, Germany

SOURCE: Justus Liebigs Annalen der Chemie (1954), 586, 110-37

CODEN: JLACBF; ISSN: 0075-4617

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 49:28006

GI For diagram(s), see printed CA Issue.

AB cf. C.A. 49, 3854f. BzH, KOH in EtOH, and EtCHO at 0° (Ger. 555,490, C.A. 26, 5102) gave 81% PhCHCMeCHO, which with MeMgBr in Et<sub>2</sub>O yielded PhCH:CMeCHMeOH, b<sub>12</sub> 135°; this distilled in vacuo with KHSO<sub>4</sub> and a small amount of p-C<sub>6</sub>H<sub>4</sub>(OH)<sub>2</sub> (I) gave PhCH:CMeCH:CH<sub>2</sub> (II), b<sub>15</sub>, 105°. With 8.4 g. maleic anhydride in 30 cc. C<sub>6</sub>H<sub>6</sub> and 0.13 g. I, 12 g. II gave 13.4 g. PhCH.CMe:CH.CH<sub>2</sub>.CH.CO.O.CO (III), m. 98°, which, heated with H<sub>2</sub>O, yielded the all-cis-dicarboxylic acid (IV), C<sub>15</sub>H<sub>16</sub>O<sub>4</sub>, m. 219°; whose Me ester, treated with an excess of 10% MeONa in MeOH, then acidified gave the all-trans isomer, m. 203°. II in Et<sub>2</sub>O with fumaroyl chloride concentrated in vacuo after 12 h., stirred with Me<sub>2</sub>CO and aqueous Na<sub>2</sub>CO<sub>3</sub>, treated with C, filtered, acidified, and extracted with Et<sub>2</sub>O gave 60% of the unstable trans isomer of IV, m. 212°

(from AcOEt-ligroine). IV (4 g.) with 48 cc. HNO<sub>3</sub> (d. 1.44) at or below 50° gave p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH(CO<sub>2</sub>H)CH(CO<sub>2</sub>H)CH(CO<sub>2</sub>H)CH<sub>2</sub>CO<sub>2</sub>H, m. 210° (decomposition). III refluxed with Ac<sub>2</sub>O and SeO<sub>2</sub> gave 85% 3-phenyl-4-methyl-phthalic anhydride, m. 163° (from ligroine). II (10 g.), 5 g. CH<sub>2</sub>:CHCO<sub>2</sub>H, and 0.1 g. I, refluxed 18 h gave a mixture, 75% of which was CMe:CH.CH<sub>2</sub>.CH<sub>2</sub>.CH(CO<sub>2</sub>H).CHPh (V), m. 133° (from AcOEt-ligroine), mixed with an unidentified oily isomer. With HNO<sub>3</sub> V gave p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH(CO<sub>2</sub>H)CH(CO<sub>2</sub>H)CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H, m. 216 (decomposition), which, heated with Ac<sub>2</sub>O, yielded p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH.CO.CH<sub>2</sub>.CH<sub>2</sub>.CHCO<sub>2</sub>H, m. 177°. V with S at 230-60°, followed by heating with aqueous Na<sub>2</sub>CO<sub>3</sub> and treatment successively with H<sub>2</sub>O<sub>2</sub>, aqueous KMnO<sub>4</sub>, and aqueous HCl gave 2,3-MePhC<sub>6</sub>H<sub>3</sub>CO<sub>2</sub>H (VI), m. 154°. The acid chloride of VI refluxed with CS<sub>2</sub> and AlCl<sub>3</sub> gave 4-methyl-9-fluorenone, yellow needles, m. 78° (from ligroine). VI warmed with aqueous Na<sub>2</sub>CO<sub>3</sub> and KMnO<sub>4</sub>, then treated with SO<sub>2</sub> and aqueous HCl, gave 2,3-(HO<sub>2</sub>C)<sub>2</sub> C<sub>6</sub>H<sub>3</sub>Ph, m. 282°, which, with concentrated H<sub>2</sub>SO<sub>4</sub> yielded 4-carboxy-9-fluorenone, m. 222°; Me ester, m. 128°. With I and HC.tplbond.CCO<sub>2</sub>H, II in PhMe gave CH:CPh.CHMe.C(CO<sub>2</sub>H):CH.CH<sub>2</sub>, m. 170° (from ligroine), whose 3,4-dihydro derivative m. 166°, and which on dehydrogenation gave VI. From MeMgBr and PhCH:CEtCHO was formed 78.8% PhCH:CEtCHMeOH, b<sub>13</sub> 140-1°, which gave 70% PhCH:CEtCH:CH<sub>2</sub> (VII), b<sub>13</sub> 104°. Prepared by methods analogous to those given above were 3-phenyl-4-ethyl-Δ<sub>4</sub>-tetrahydrophthalic anhydride, m. 82°; corresponding all-cis acid (VIII), m. 206° (decomposition), which, when heated 20 min. at 280°, gave 20% of the neo-cis isomer, m. 158°. The di-Me ester of VIII with MeONa gave the all-trans isomer (IX) of VIII, m. 184°. With fumaroyl chloride, VII gave the "unstable" trans isomer of VIII, m. 203°, which with MeONa yielded IX. VIII with HNO<sub>3</sub> formed p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH(CO<sub>2</sub>H)CH(CO<sub>2</sub>H)CH<sub>2</sub>CO<sub>2</sub>H. 3,4-PhMeC<sub>6</sub>H<sub>3</sub>CO<sub>2</sub>H m. 183°; anhydride, m. 81°. CH<sub>2</sub>:CHCO<sub>2</sub>H, VII, and I in PhMe gave a mixture of the relatively little soluble (predominating) cis-2-phenyl-3-ethyl-Δ<sub>3</sub>-tetrahydrobenzoic acid (X), m. 144°, and the trans isomer (XI), m. 99°, separated by fractionation from AcOEt-ligroine. XI was formed by heating X at 280°, or from the Me ester of X with MeONa. X with HNO<sub>3</sub> gave p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH(CO<sub>2</sub>H)CH(CO<sub>2</sub>H)CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H, m. 216°. X in AcOEt with O<sub>3</sub> followed by H<sub>2</sub>O, a few drops of AcOH, and H<sub>2</sub>O<sub>2</sub> gave HO<sub>2</sub>CCH<sub>2</sub>CH<sub>2</sub>CH(CO<sub>2</sub>H)CHPhCOEt, m. 151°. Dehydrogenation of X gave 2,3-PhEtC<sub>6</sub>H<sub>3</sub>CO<sub>2</sub>H (XII), m. 159°, yielding 4-ethyl-9-fluorenone, m. 57°, on cyclization. CH.tplbond.CCO<sub>2</sub>H and VII in PhMe gave CH:CEt.CHPh.C(CO<sub>2</sub>H):CH.CH<sub>2</sub> (XIIa), forming XII on dehydrogenation. The aldehyde analog of XIIa, b<sub>22</sub> 183-4° (semicarbazone, C<sub>16</sub>H<sub>19</sub>ON<sub>3</sub>, m. 228°), was prepared from VII and CH.tplbond.CCHO, and, when oxidized, gave XIIa. To 370 g. EtOH, 15 g. KOH, and 210 g. BzH was added dropwise, 210 g. PhCH<sub>2</sub>CHO at (or below) 10°, giving 250 g. PhCH:CPhCHO, b<sub>14</sub> 190°, m. 94°. The following were formed by methods analogous to those described: PhCH:CPhCHMeOH, b<sub>4</sub> 164°, and (incompletely purified and unstable) PhCH:CPhCH:CH<sub>2</sub> (XIII), m. 57° (from MeOH); 3,4-diphenyl-Δ<sub>4</sub>-tetrahydrophthalic anhydride, m. 157°, whose corresponding all-cis-acid, C<sub>20</sub>H<sub>18</sub>O<sub>4</sub>, m. 206°, was converted into the neo-cis-isomer, m. 164°, on heating. With fumaroyl chloride, XIII gave the trans-isomer (XIV) (from ligroine), m. 218°, also formed by rearrangement of the all-cis and neo-cis isomers. The anhydride of XIV m. 157°. 3,4-Diphenylphthalic acid m. 200° (decomposition); anhydride, m. 150°. XIII (92 g.) with

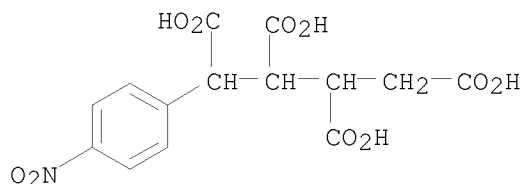
CH<sub>2</sub>:CHCO<sub>2</sub>H in C<sub>2</sub>H<sub>6</sub> gave a mixture of 9 g. cis- and 3 g. trans-CH:CHPh.CHPh.CH(CO<sub>2</sub>H).CH<sub>2</sub>.CH<sub>2</sub>, m. 176° and 148°, resp., the cis readily passing into the trans form. When this reaction was carried out in PhMe equal parts of the 2 isomers were formed. Either isomer heated with S or SeO<sub>2</sub> gave 2,3-Ph<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CO<sub>2</sub>H (XIV), m. 161°; 4-phenyl-9-fluorenone, m. 112°. The 2,5-dihydro derivative, m. 182°, of XIV was readily dehydrogenated to XIV. 2,3-Diphenyl-2,5-dihydrobenzaldehyde b15 210-15° (semicarbazone, m. 229°). BzH (2 mol) refluxed 2 h. with 1 mol (CH<sub>2</sub>:CHCO)<sub>2</sub>O and Et<sub>3</sub>N gave 37% CH<sub>2</sub>:CHC(CO<sub>2</sub>H):CHPh (XV), m. 92°; Me ester, b12 144-5°, has a fruity odor. In xylene XV refluxed 20 h. with maleic anhydride and small amts. of di-MeCHPh derivative of I (as stabilizer) gave 89% 3-phenyl-4-carbomethoxy-Δ<sup>4</sup>-tetrahydrophthalic acid (XVI), m. 184-5°, giving, on saponification, the 4-carboxy analog (XVIa), m. 246-7°. The all-cis-tri-Me ester of XVIa, C<sub>18</sub>H<sub>20</sub>O<sub>6</sub>, m. 137-8°, was converted by NaOMe or by heating with concentrated HCl, followed by reesterification, into the all-trans isomer (XVII), m. 110° (from ligroine). Ozonization of XVIa gave PhCH(CO<sub>2</sub>H)CH(CO<sub>2</sub>CH)CH(CO<sub>2</sub>H)CH<sub>2</sub>CO<sub>2</sub>H, m. 196-7° (from MeCN); tetra-Me ester, m. 102-3° (from petr. ether). XV refluxed 20 h. in xylene with di-Me fumarate gave the unstable trans-CH:C(CO<sub>2</sub>Me).CHPh.CH(CO<sub>2</sub>Me).CH(CO<sub>2</sub>Me).CH<sub>2</sub>, m. 76-7° (from petr. ether), converted by MeONa into XVII. XV and CH<sub>2</sub>:CHCO<sub>2</sub>H, followed by methylation with Me<sub>2</sub>SO<sub>4</sub>, gave CH:C(CO<sub>2</sub>Me).CHPh.CH(CO<sub>2</sub>Me).CH<sub>2</sub>.CH<sub>2</sub>, b0.005 129-32°, m. 67-8° (from ligroine); the free acid (XVIII), m. 236-7°, treated with Br at 200°, followed by oxidation in aqueous Na<sub>2</sub>CO<sub>3</sub> with KMnO<sub>4</sub>, a few drops of aqueous NaHSO<sub>3</sub>, digestion on the steam bath with 3 % Na-Hg, and acidification with HCl, gave 2,6-(HO<sub>2</sub>C)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>Ph, m. 280-1°. Under N at 235°, XVIII in quinoline with Cu chromite gave, after esterification, PhCH.CH(CO<sub>2</sub>Me).CH<sub>2</sub>.CH<sub>2</sub>.CH:CH, b13 154-6°; free acid, m. 97-8°. XVIII shaken 7 h. in MeOH with Raney Ni at 250 atmospheric and 210° followed by treatment with CH<sub>2</sub>N<sub>2</sub> gave the di-Me ester of the dihydro derivative of XVIII, yellow oil; dianilide, m. 328-30°. XVIII, ozonized in AcOEt (followed by a fully described series of purifications), gave HO<sub>2</sub>CCHPhCH(CO<sub>2</sub>H)CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H, m. 194-5° (from MeCN or AcOEt). PhCH<sub>2</sub>CHO (220 g.), 93 g. AcH, 370 g. 50% EtOH, and 93 g. crystalline AcONa, 24 h. at 110-20°, gave 116 g. MeCH:CPhCHO, b13, 117°; this, in Et<sub>2</sub>O with MeMgBr (from 25 g. Mg) at 0°, followed by standing 2 h. at room temperature and final heating with aqueous

NH<sub>4</sub>Cl,

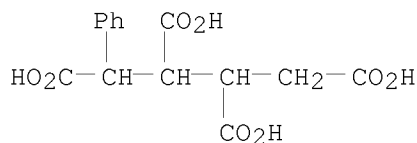
gave 104 g. MeCH:CPhCHMeOH, b13 118°, which with KHSO<sub>4</sub> yielded 78% MeCH:CPhCH:CH<sub>2</sub> (XIX), b12 72-3°. By reactions analogous to those given, XIX gave 3-methyl-4-phenyl-Δ<sup>4</sup>-tetrahydrophthalic anhydride, m. 167°, and 3,4,2-(HO<sub>2</sub>C)<sub>2</sub>MeC<sub>6</sub>H<sub>2</sub>Ph, m. 184-5° (decomposition). XIX with CH<sub>2</sub>:CHCO<sub>2</sub>H gave an unsepd. oily mixture of isomeric acids which, after methylation and dehydrogenation with S, formed a mixture of esters, b13 186-90°; this, after saponification, gave about 4 parts of the less soluble 2,3-MePhC<sub>6</sub>H<sub>3</sub>CO<sub>2</sub>H (XX), m. 160°, and 1 part of 3,4-MePhC<sub>6</sub>H<sub>3</sub>CO<sub>2</sub>H (XXI), m. 146°. On oxidation XX gave 2,3-(HO<sub>2</sub>C)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>Ph, m. 185° (anhydride, m. 150-1°), and XXI yielded 2,4-(HO<sub>2</sub>C)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>Ph, m. 245-6°. 2-Carboxy-9-fluorenone sublimes above 335°; Me ester, m. 181°. With HC.tplbond.CCO<sub>2</sub>H, XIX formed a mixture (m. 151°) of CH:CPh.CHMe.C(CO<sub>2</sub>H):CH.CH<sub>2</sub> and CH:CPh.CHMe.CH:C(CO<sub>2</sub>H).CH<sub>2</sub>, which yielded XX and XXI on dehydrogenation. XIX in Et<sub>2</sub>O saturated with SO<sub>2</sub>, heated 10 h. in a bomb tube at 100° and cooled to -20°, gave the sulfone of XIX, m. 93°, decomposing

into XIX and SO<sub>2</sub> at 144°. In C<sub>6</sub>H<sub>6</sub>, this sulfone reacted at 150° with maleic anhydride, CH<sub>2</sub>:CHCO<sub>2</sub>H, or CH.tplbond.CCO<sub>2</sub>H, giving products in each case identical with those formed by the parent XIX. 27 refs.

IT 854462-26-3P, 1,2,3,4-Butanetetracarboxylic acid,  
1-(p-nitrophenyl)- 875851-27-7P, 1,2,3,4-Butanetetracarboxylic  
acid, 1-phenyl-  
RL: PREP (Preparation)  
(preparation of)  
RN 854462-26-3 HCAPLUS  
CN 1,2,3,4-Butanetetracarboxylic acid, 1-(4-nitrophenyl)- (CA INDEX NAME)



RN 875851-27-7 HCAPLUS  
CN 1,2,3,4-Butanetetracarboxylic acid, 1-phenyl- (CA INDEX NAME)



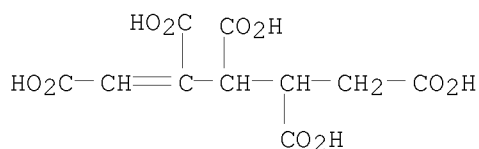
L7 ANSWER 9 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN  
ACCESSION NUMBER: 1955:6701 HCAPLUS  
DOCUMENT NUMBER: 49:6701  
ORIGINAL REFERENCE NO.: 49:1368d-f  
TITLE: Aconitate-fumarate adducts  
INVENTOR(S): Dazzi, Joachim  
PATENT ASSIGNEE(S): Monsanto Chemical Co.  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2687428		19540824	US 1951-228113	19510524 <--

AB Addition products (I) of dialkyl fumarates and trialkyl aconitates (II) are plasticizers for polyvinyl chlorides. They range from clear, viscous liquids to waxy solids, and have the general formula CH(CO<sub>2</sub>Y):C(CO<sub>2</sub>Y')CHCO<sub>2</sub>Y''CHCO<sub>2</sub>RCH<sub>2</sub>CO<sub>2</sub>R', in which Y, Y', Y'', R and R' are alkyl radicals of from 1 to 4 C atoms. In an example triethyl aconitate 258.1 g. and diethyl fumarate (III) 342.76 g. are heated at reflux temperature (220°) for 3 hrs. and at 201-190° for 15 hrs. After a

low-boiling fraction is distilled off, the rest of the mixture is refluxed at 249° for an addnl. 12 hrs. The final I is probably pentaethyl 1-pentene-1,2,3,4,5-pentacarboxylate, b0.5 200-20°. nD25 1.4690, a free acid content 2.99%. In U.S. 2,687,429, instead of II dialkyl succinates are used. Thus, diethyl succinate 87.1 g. and III 258.3 are refluxed at 220-45° for 133 hrs. Fractionation gave probably tetraethyl 1,2,3,4-butanetetracarboxylate, b0.3-0.5 220-2°, nD25 1.4648, free acid value 0.41. Vinyl resins plasticized with I possess low-temperature flexibility and low volatility.

IT 855443-74-2, 1-Pentene-1,2,3,4,5-pentacarboxylic acid  
(pentaalkyl esters, as plasticizers for polyvinyl chlorides)  
RN 855443-74-2 HCAPLUS  
CN 1-Pentene-1,2,3,4,5-pentacarboxylic acid (CA INDEX NAME)



L7 ANSWER 10 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1953:58670 HCAPLUS

DOCUMENT NUMBER: 47:58670

ORIGINAL REFERENCE NO.: 47:9979a-f

TITLE: Veratrum alkaloids. II. The constitution of the hexanetetracarboxylic acid from cevine and germin

AUTHOR(S): Elming, N.; Vogel, Ch.; Jeger, O.; Prelog, V.

CORPORATE SOURCE: Eidg. Tech. Hochschule, Zurich, Switz.

SOURCE: Helvetica Chimica Acta (1952), 35, 2541-8

CODEN: HCACAV; ISSN: 0018-019X

DOCUMENT TYPE: Journal

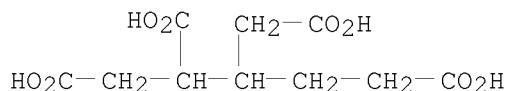
LANGUAGE: German

GI For diagram(s), see printed CA Issue.

AB cf. ibid. 838. Oxidation of 70 g. cevine with CrO3 and H2SO4 according to Craig and Jacobs (C.A. 33, 7804.8), esterification of the crude acids, and fractional distillation give 7.4 g. tetra-Me hexanetetracarboxylate (I), b0.15 150-64°, m. 65-6°, [α]D 21° (c 0.97, CHCl3). Heating I 7 hrs. with 0.5N alc. KOH gives the free acid (II), m. 155-8° (decomposition), [α]D 29° (c 0.66, Me2CO). I and II show no MeC group by the Kuhn-Roth method. Heating 150 mg. II at 200-50° and 10 mm. gives 50% ketone anhydride, C9H10O4 (III), m. 117-18°, [α]D 130° (c 0.62, Me2CO). The infrared absorption spectrum (IR) of III indicates that the CO group is in a 5-membered ring. Treating 300 mg. III with 100 mg. Na in 5 cc. EtOH, dissolving the Na salt formed by the addition of a few drops of H2O, and treating the mixture with 200 mg. BzH 4 days at 20° give 300 mg. benzylideneketo carboxylic acid, C16H16O5 (IV), m. 195-6°, [α]D -41° (c 0.85, MeCO). IV shows an ultraviolet absorption curve similar to that of benzylidenecyclopentanone, indicating that III is a cyclopentanone derivative. Because in the IR of III the band (1850-1880 cm.-1) typical for cyclic 5-membered acid anhydrides is missing, the acid anhydride ring in III must have more than 5 ring members. Of the 7 possible constitutional formulas suggested for I, that of

4-carboxymethyl-1,3,7-heptanetricarboxylic acid, HO<sub>2</sub>CCH<sub>2</sub>CH<sub>2</sub>CH(CO<sub>2</sub>H)CH(CH<sub>2</sub>CO<sub>2</sub>H)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H, is preferred and its structure can be located only in the rings A, B, C, and D as in (Va and b), and not in D and E.

IT 875226-88-3P, 1,2,5-Pentanetricarboxylic acid, 3-(carboxymethyl)-  
 RL: PREP (Preparation)  
 (preparation of)  
 RN 875226-88-3 HCAPLUS  
 CN 1,2,5-Pentanetricarboxylic acid, 3-(carboxymethyl)- (CA INDEX NAME)



L7 ANSWER 11 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1953:9085 HCAPLUS

DOCUMENT NUMBER: 47:9085

ORIGINAL REFERENCE NO.: 47:1604d-g

TITLE: Syntheses with dicarboxylic acids. III. Malonic acid ester syntheses with  $\alpha$ -bromoadipic esters

AUTHOR(S): Treibs, Wilhelm; Mayer, Roland

CORPORATE SOURCE: Univ. Leipzig, Germany

SOURCE: Chemische Berichte (1952), 85, 612-15

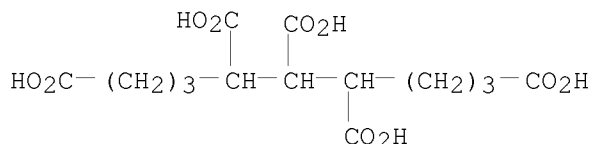
CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB Polycarboxylic acids are prepared by condensation of  $\alpha$ -bromoadipic acid (I) with CH<sub>2</sub>(CO<sub>2</sub>Et)<sub>2</sub> (II). Adding 0.2 mol. II and then 0.2 mol. di-Et  $\alpha$ -bromoadipate (III) to 100 cc. EtOH containing 0.2 mol. Na, refluxing the mixture 3 hrs. on a water bath, pouring it into 0.6 mol. 1% HCl, extracting immediately with ether, and fractionally distilling the residue of the ether extract give 50% tetra-Et 1,2,3,5-pentanetetracarboxylate (IV), RO<sub>2</sub>C(CH<sub>2</sub>)<sub>3</sub>CH(CO<sub>2</sub>R)CHR'CO<sub>2</sub>R (IVa, R = Et, R' = CO<sub>2</sub>Et), b<sub>10</sub> 200-2°, b<sub>25</sub> 227-8°, n<sub>D</sub>18 1.4451, d<sub>4</sub>18 1.1035. Heating IV with 5 times its volume concentrated HCl 3 hrs. on a water bath and evaporating the solution in vacuo give the free acid (V), viscous oil which, on standing in a refrigerator 2 weeks, crystallizes to minute needles, m. 117-18° (decomposition, CO<sub>2</sub> evolution). Heating V at 125-30° until the evolution of CO<sub>2</sub> ceases gives 1,2,5-pentanetricarboxylic acid (IVa, R = R' = H), m. 85-6°. Condensation of I with NCCH<sub>2</sub>CO<sub>2</sub>Et gives 55% tri-Et 1-cyano-1,2,5-pentanetricarboxylate (IVa, R = Et, R' = CN), b<sub>16</sub> 215-19°, n<sub>D</sub>14 1.4537, d<sub>4</sub>14 1.1247. Adding dropwise the calculated amount Br to 36 g. IV in CHCl<sub>3</sub> and distilling the residue of the CHCl<sub>3</sub> solution in vacuo give 90% 1-Br analog, b<sub>12</sub> 235-42°. Treating 0.1 mol. IV with EtONa, condensing the mono-Na compound with III 15 hrs. on a water bath, and working up the product in the usual way give 25% hexa-Et 1,4,5,5,6,9-nonanehexacarboxylate (VI), RO<sub>2</sub>CCR'[CH(CO<sub>2</sub>R)](CH<sub>2</sub>)<sub>3</sub>CO<sub>2</sub>R (VIa, R = Et, R' = CO<sub>2</sub>Et), viscous yellow oil, b<sub>0.03</sub> 243-8°, n<sub>D</sub>18 1.4921, d<sub>4</sub>18 1.1932. VI is also obtained in 10% yield when 0.1 mol. CNa<sub>2</sub>(CO<sub>2</sub>Et)<sub>2</sub> is heated in 100 cc. EtOH with 0.2 mol. III 60 hrs. on a water bath. Refluxing VI with 6 times its volume concentrated HCl 4 hrs. gives 1,4,5,6,9-nonanepentacarboxylic

acid (VIa, R = R' = H), rosettes, m. 124-6°.  
 IT 859987-87-4P, 1,4,5,6,9-Nonanepentacarboxylic acid  
 RL: PREP (Preparation)  
 (preparation of)  
 RN 859987-87-4 HCAPLUS  
 CN 1,4,5,6,9-Nonanepentacarboxylic acid (CA INDEX NAME)



L7 ANSWER 12 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

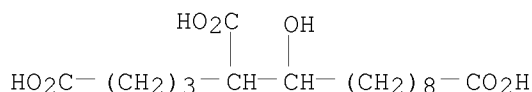
ACCESSION NUMBER: 1951:29562 HCAPLUS  
 DOCUMENT NUMBER: 45:29562  
 ORIGINAL REFERENCE NO.: 45:5106c-g  
 TITLE: The extension of the Reformatskii synthesis  
 AUTHOR(S): Treibs, Wilhelm; Leichssenring, Gert  
 CORPORATE SOURCE: Univ. Leipzig, Germany  
 SOURCE: Chemische Berichte (1951), 84, 52-5  
 CODEN: CHBEAM; ISSN: 0009-2940  
 DOCUMENT TYPE: Journal  
 LANGUAGE: Unavailable

AB By applying the Reformatskii reaction to mono- and dihalogenated dicarboxylic acids, mono- and disubstituted dicarboxylic acids which may be of physiol. interest are obtained. Treating 1 mol. (CH<sub>2</sub>CH<sub>2</sub>COC1)<sub>2</sub>, b18 130-3°, with 4 mols. Br in the presence of 10 g. red P, dropping the mixture into 600 cc. absolute MeOH, and fractionating the mixture give 120

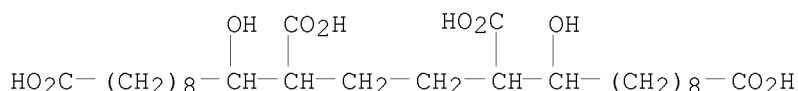
g.

di-Me  $\alpha$ -bromoadipate (I), b14 155-7°, d204 1.4101, n20D 1.4639, and 30 g. di-Me  $\alpha,\delta$ -dibromoadipate (II), b14 182-4°. Di-Me  $\alpha$ -bromoazelaate (III), prepared in a similar way, b12 180°, d204 1.2351, n20D 1.4635; di-Me  $\alpha,\eta$ -dibromoazelaate (IV), b12 212°, d204 1.4937, n20D 1.4919. Ozonization of 40 g. Et 11-hendecenoate in AcOH at CO<sub>2</sub>-Me<sub>2</sub>CO temperature gives 20 g. Et c-oxocaprinate (V), b12 162-3°, d204 0.9611, n204 1.4470. Heating 1/6 mol. C<sub>7</sub>H<sub>15</sub>CHO (VI) and 1/6 mol. I with 11 g. Zn tinsels 1 hr. on a water bath, adding ice NH<sub>4</sub>Cl, and saponifying the product with 10% NaOH/MeOH give 3 g.  $\alpha$ -octylideneadipic acid, C<sub>7</sub>H<sub>15</sub>CH:C(CO<sub>2</sub>H)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H, b12 220-40°, crystals from C<sub>6</sub>H<sub>6</sub>, m. 94-5°. In the same way 0.2 mol. VI and 0.1 mol. II give 3 g.  $\alpha,\delta$ -dioctylideneadipic acid, [CH<sub>2</sub>C ( : CHC<sub>7</sub>H<sub>15</sub>) CO<sub>2</sub>H]<sub>2</sub>, b12 250-60°, m. 73-5°. VI (19 g.) and III give 1 g.  $\alpha$ -octylideneazelaic acid, viscous oil, b12 263-7°.  $\alpha,\eta$ -Dioctylideneazelaic acid, prepared from IV and VI, b12 275-8°. Treating 0.1 mol. V and 0.1 mol. I in 100 cc. C<sub>6</sub>H<sub>6</sub> with 6.5 g. Zn tinsels 0.5 hr. at 60-5° and saponifying the crude ester give 5-hydroxy-1,4,13-tridecanetricarboxylic acid, HO<sub>2</sub>C(CH<sub>2</sub>)<sub>8</sub>CH(OH)CH(CO<sub>2</sub>H)(CH<sub>2</sub>)<sub>3</sub>CO<sub>2</sub>H, b12 250-60°, m. 125-6°. V and 11 g. II give 4 g. 9-14-dihydroxy-1,10,13,22-docosanetetracarboxylic acid, [HO<sub>2</sub>C(CH<sub>2</sub>)<sub>8</sub>CH(OH)CH(CO<sub>2</sub>H)CH<sub>2</sub>]<sub>2</sub>, b12 270-80°, m. 130-2°.

IT 858788-83-7P, 1,4,13-Tridecanetricarboxylic acid, 5-hydroxy-  
 859310-02-4P, 1,10,13,22-Docosanetetracarboxylic acid,  
 9,14-dihydroxy-  
 RL: PREP (Preparation)  
 (preparation of)  
 RN 858788-83-7 HCAPLUS  
 CN 1,4,13-Tridecanetricarboxylic acid, 5-hydroxy- (CA INDEX NAME)



RN 859310-02-4 HCAPLUS  
 CN 1,10,13,22-Docosanetetracarboxylic acid, 9,14-dihydroxy- (CA INDEX NAME)



L7 ANSWER 13 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1947:11808 HCAPLUS  
 DOCUMENT NUMBER: 41:11808  
 ORIGINAL REFERENCE NO.: 41:2395b-i,2396a  
 TITLE: Vinyl polymers. XXIV. The reaction of benzoyl peroxide  
 and maleic ester  
 AUTHOR(S): Marvel, C. S.; Prill, E. J.; DeTar, D. F.  
 CORPORATE SOURCE: Univ. of Illinois, Urbana  
 SOURCE: Journal of the American Chemical Society (1947  
 ), 69, 52-8  
 CODEN: JACSAT; ISSN: 0002-7863  
 DOCUMENT TYPE: Journal  
 LANGUAGE: Unavailable  
 AB cf. C.A. 40, 4685.6. A mixture of maleic ester (I) and 0.33-0.5 mole Bz2O2  
 was kept at 55° 170-700 hrs.; in most expts. p-dioxane (400 cc. per  
 mole of ester) was used as the solvent. Variations in the conditions of  
 the reactions had no significant effect on the yield or the proportions of  
 products formed. The solvent was removed under slightly reduced pressure  
 in a current of air, the Bz2O2 destroyed with NaI in Me2CO, the residue in  
 ether washed with aqueous NaHCO3, and the products distilled until the  
 fractions  
 crystallized or could be hydrolyzed to crystalline acids. The recovered I  
 varied  
 from 3 to 20%. The fraction b1 105-18° yielded 5%  
 PhCH(CO2Me)CH2CO2Me, m. 54.5-5.5°; the portion with low n on alkaline  
 hydrolysis yielded p-dioxan-2-ylsuccinic acid (II), C8H12O6, m.  
 126-8°; II results also on hydrolysis of a fraction b4  
 187-8°, nD20 1.4578, from the reaction with (:CHCO2Et)2. A  
 fraction b1 182-95° gave a Me ester, C18H20O8, m. 149.4-9.8°  
 (corrected), of an acid, C14H12O8 (III), m. 236°; the Et ester m.  
 77-8°; a fraction b1 190-200° gave an isomer (IV) of III, m.  
 222° (decomposition); Me ester m. 95-6°. III and IV (yields of



25%) are probably stereoisomeric forms of tetra-Me 1,2,3,4-tetralintetracarboxylate or tetra-Me 1-methylhydrindenetetracarboxylate. The fractions b1 190-200° from the reactions in dioxane analyze for dioxane-containing compds. The fraction b1 140-4° yielded about 1% of an acid C10H12O8, m. 185-7°; both acid and ester decolorize KMnO4 solution. The reaction products in CCl4 included C2Cl6, PhCl, and di-Et  $\alpha,\beta$ -bis(trichloromethyl)succinate (IVA), m. 93.5-4.5°.

(EtO2C)2CHCH2CO2Et (42 g.) and 3.76 g. Na in 100 cc. absolute EtOH, treated at 0° with 20 g. 2-chloro-p-dioxane, stirred 2 hrs., and refluxed 2 hrs., give 15 g. of the di-Et  $\alpha$ -(p-dioxan-2-yl)- $\alpha$ -carbethoxysuccinate (V), b1 150-2°, nD20 1.4530; a by-product is ethoxydioxane, b50 81-2°, nD20 1.4258. Hydrolysis of V with 20% KOH (refluxing 9 hrs.) gives 70% II; di-Me ester (VI) b1 113-14°, nD20 1.4592. VI with 5 vols. concentrated NH4OH, allowed to stand 1 week and the product crystallized from EtOH, gives dioxanylsuccinamic acid, m. 160-1°, and p-dioxan-2-ylsuccinamide, m. 234-5° (decomposition), sublimes 220°/1 mm. II, refluxed 1 hr. with AcCl, gives the anhydride, m. 121-3°. III on oxidation with alkaline KMnO4 gives 22% o-C6H4(CO)2O; refluxed with Ac2O 5 min., III yields a dianhydride, C14H8O6, m. 192.7-4.4° (corrected). Oxidation of IV gives 38% o-C6H4(CO)2O. Tests of III for unsatn. were neg.; attempts to dehydrogenate III did not give definite products. IVA on hydrolysis with 70% H2SO4 gives (CH2CO2H)2; refluxed 12 hrs. with 20% KOH, IVA yields  $\alpha,\beta$ -bis(dichloromethylene)succinic acid (?), m. 219° (decomposition). EtO2CCH2CH(CO2Et)CH(CO2Et)2 (107.5 g.) in 100 cc. absolute

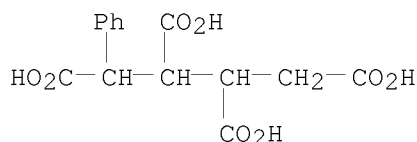
EtOH

containing 6.7 g. Na, treated after 0.5 hr. with 70 g. PhCHBrCO2Et and the mixture boiled 5 hrs., gives 31 g. 1-phenyl-1,2,2,3,4-pentacarbethoxybutane, b1 195-200°, nD20 1.4913; 22.5 g. ester in 250 cc. 20% KOH, refluxed 32 hrs., gives 1-phenyl-1,2,3,4-butanetetracarboxylic acid, m. 202-4°; the residue yields an acid, m. 167.5-8.7°. The higher-melting acid yields a tetra-Me ester, m. 142.5-3.5°; the other acid yields an ester, m. 117-18°; these appear to be 2 of the 4 possible racemic forms. These esters are appreciably more soluble in H2O than the esters of III and IV and yield BzOH on oxidation.

IT 875851-27-7, 1,2,3,4-Butanetetracarboxylic acid, 1-phenyl- (isomers)

RN 875851-27-7 HCAPLUS

CN 1,2,3,4-Butanetetracarboxylic acid, 1-phenyl- (CA INDEX NAME)



L7 ANSWER 14 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1943:27195 HCAPLUS

DOCUMENT NUMBER: 37:27195

ORIGINAL REFERENCE NO.: 37:4364d-h

TITLE: Formation of the oxide bridge at the double bond

AUTHOR(S): Loginov, P. V.

SOURCE: Trudy Voroshilov. Gosudarst. Pedagog. Inst. (

1940), 2, 213-22

From: Khim. Referat. Zhur 4, No. 3, 28-9(1941).

DOCUMENT TYPE:

Journal

LANGUAGE:

Unavailable

GI For diagram(s), see printed CA Issue.

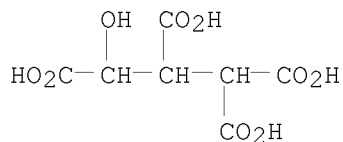
AB Tri-Et butyrolactonetetracarboxylate was obtained from the reaction of di-Me dl-trans-ethyleneoxidedicarboxylate with Na malonic ester: Ba butyrolactonetetracarboxylate was obtained by emulsifying the ester with Ba(OH)<sub>2</sub>. The free hydroxytricarboxylic acid was obtained from Ba butyrolactonetetracarboxylate by the reaction with H<sub>2</sub>SO<sub>4</sub>. The acid liberated 1 mol. of CO<sub>2</sub> at 70-90° and was transformed almost quantitatively into the dicarboxylic acid: The tri-Me  $\alpha,\beta,\gamma$ -butyrolactonetetracarboxylate, a light sirup, is insol. in water and soluble in ether, b<sub>10</sub> 202°, d<sub>420</sub> 1.2180, n<sub>D20</sub> 1.4534. The  $\alpha,\beta,\gamma$ -butyrolactonetetracarboxylic acid is a very hygroscopic white amorphous product, b. 58-60° (slowly), is soluble in water, ether, alc. and other organic solvents, insol. in petr. ether.

It forms NH<sub>4</sub> and Ag salts.

IT 751454-84-9, 1,1,2,3-Propanetetracarboxylic acid, 3-hydroxy- (and  $\gamma$ -lactone and derivs.)

RN 751454-84-9 HCAPLUS

CN 1,1,2,3-Propanetetracarboxylic acid, 3-hydroxy- (9CI) (CA INDEX NAME)



L7 ANSWER 15 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1941:4513 HCAPLUS

DOCUMENT NUMBER: 35:4513

ORIGINAL REFERENCE NO.: 35:731e-i,732a-h

TITLE: Mechanism of polymerization. VI. Heat polymerization of methyl sorbate and the constitution of the dimeric products

AUTHOR(S): Farmer, Ernest Harold; Morrison-Jones, Colin R.

SOURCE: Journal of the Chemical Society (1940)

1339-46

CODEN: JC SOA9; ISSN: 0368-1769

DOCUMENT TYPE:

Journal

LANGUAGE:

Unavailable

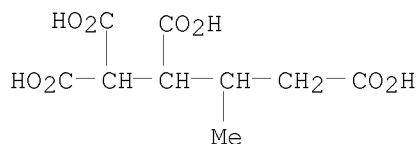
AB cf. C. A. 34, 7863.3. Polymerization of Me sorbate (I) occurs readily when it is

heated in CO<sub>2</sub> at atmospheric pressure between its b. p. (180°) and 230°; after 2.5 or 3.5 h., there result 14 or 6% of monomeric material (b<sub>3</sub> below 110°) (II), 75 or 81% of dimeric material (b<sub>3</sub>, 110-50°) (III) and 10 or 12% of a viscous oily material, mostly trimeric (this was not investigated). II consists mainly of I but was not entirely homogeneous. III is a complex mixture of isomers, systematic distillation of which at 3.5 mm. under jacketed Dufton-type columns fitted for close reflux control gives 2 constant-boiling mixts., IV (13% of III), d<sub>418</sub>

1.062, nD<sub>18</sub> 1.47806 and V (12% of III), nD<sub>17</sub> 1.49063. These give some proportion of solid acid on hydrolysis. The main portion of the III has not been investigated. Catalytic reduction of IV (PtO<sub>2</sub>) for 8 h. gives a tetrahydride, C<sub>14</sub>H<sub>24</sub>O<sub>4</sub>, b<sub>0.1</sub> 82-2.5°, nD<sub>17</sub> 1.4598, d<sub>417</sub> 1.034. Hydrolysis of IV by heating with 10 parts of MeOH-KOH for 20-30 min. gives a solid K salt, yielding 5-9% (based on IV) of an acid (VI), m. 200-5°; the filtrate yields a pale yellow resinous acid which, crystallized from C<sub>6</sub>H<sub>6</sub>-petr. ether, gives 12-15% of an acid (VII), m. 165-85°; the major portion (75-80%) is a semi-resinous acid mixture which has not been studied. Recrystn. of VI (5 times with 50% loss) from aqueous AcOH or aqueous MeOH gives 1-methyl-2-propenyl-4-cyclohexene-3,4-dicarboxylic acid (VIII), C<sub>12</sub>H<sub>16</sub>O<sub>4</sub>, m. 216°; the residues, crystallized from ether-petr. ether, give the isomeric 1-methyl-3-propenyl-4-cyclohexene-2,4-dicarboxylic acid (IX), m. 200°. Recrystn. of VII from AcOEt or ether-petr. ether gives 1-methyl-3-propenyl-4-cyclohexene-2,4-dicarboxylic acid (X), m. 191°; the residues yield an acid, m. 164-9°, which could not be purified further and whose constitution was not established. Hydrolysis of V with MeOH-KOH gives some VIII but the nature of the major portion of the acids was not determined. V and PhNHMgBr give a mixture of dianilides; the solid portion m. 288-90° (decomposition); this could not be hydrolyzed to the acid. The structures of the above acids are based on the following reactions. Catalytic reduction of VIII gives 1-methyl-2-propylcyclohexane-3,4-dicarboxylic acid, m. 188°, and a 2nd acid, m. 154-9° (which was not studied). Refluxing VIII with AcCl for 6 h. gives an anhydride, C<sub>12</sub>H<sub>14</sub>O<sub>3</sub>, m. 84°, which gives VIII on boiling with H<sub>2</sub>O for a few min. Dehydrogenation of VIII by heating with an equal weight of Se in CO<sub>2</sub> for 18 h. or by heating with 5% Pd-C at 290-305° for 6 h. gives the acid C<sub>12</sub>H<sub>14</sub>O<sub>4</sub>, m. 178°, oxidation of which with aqueous KMnO<sub>4</sub> gives mellophanic acid (XI); in 1 experiment the crude Se dehydrogenation product was extracted with Et<sub>2</sub>O and the extract refluxed with Na; the Na salt which separated yields an acid m. 167-9°, oxidation of which gives an acid m. 212-14°; crystallization of the Me ester from MeOH gives the Me ester of XI and Me 3-oxalylbenzene-1,2,4-tricarboxylate, m. 102°. Oxidation with 5% KMnO<sub>4</sub> of the reaction product of VIII with O<sub>3</sub> in AcOEt gives 0.25 mol of AcOH, a trace of (CO<sub>2</sub>H)<sub>2</sub> and some 2-methyl-1,3,4-butanetricarboxylic acid (XII); oxidation of the solid ozonide formed in CHCl<sub>3</sub> gives 0.61 mol of AcOH, a trace of (CO<sub>2</sub>H)<sub>2</sub> and 0.5 mol of 2-methyl-1,3,4,4-butanetetracarboxylic acid, m. 160° with loss of CO<sub>2</sub> and formation of XII. Oxidation of VIII with KMnO<sub>4</sub> gives (CO<sub>2</sub>H)<sub>2</sub> progressively throughout the reaction; this and AcOH are the only products formed; if the oxidation is interrupted and the intermediate products heated in acid solution much (CO<sub>2</sub>H)<sub>2</sub> is formed by hydrolysis. Catalytic reduction of X gives 4-methyl-2-propylphthalic acid (XIII), m. 164°, and a 2nd acid, m. 145-50° (not examined). X does not yield an anhydride with AcCl. Dehydrogenation yields m-MeC<sub>6</sub>H<sub>4</sub>Pr which is oxidized by KMnO<sub>4</sub> to isophthalic acid. The action of O<sub>3</sub> and KMnO<sub>4</sub> on X gives the same products as with VIII. Catalytic reduction of IX gives XIII; IX is not isomerized by heating with concentrated HCl at 160° for 10 h. With regard to the mechanism of the formation of the cyclohexenic dimers the striking feature of the results is the addition of the CH:CHCO group of 1 reacting member across the terminals of the usually strongly polarized diene system MeCH:CHCH:CH in both of the possible directions. It is not surprising, in view of the ease with which Diels-Alder reactions are normally effected, that the CH:CHCO group rather than the MeCH:CH group of sorbic acid

functions as the reactive part of the addendum mol. but the results make it clear that if the initial mol. activations are strictly polar in character then both of the polarized forms  $\text{MeC}^+\text{H}:\text{CHCH}:\text{C}-\text{HCO}$  and  $\text{MeC}-\text{H}:\text{CHCH}:\text{C}^+\text{HCO}$  must be available at the moment of reaction and participate in the addition. It seems likely that the course of the reaction in this thermal polymerization is dependent on the formation of free-radical forms of the monomeric mols. rather than polar ones. It is possible that other dimeric forms derived from the addition of the  $\text{MeCH}:\text{CH}$  unit of 1 sorbic acid mol. to the terminals of a 2nd mol. are contained in the unidentified portion of the polymerizate.

IT 855242-80-7P, 1,1,2,4-Butanetetra-carboxylic acid, 3-methyl-  
 RL: PREP (Preparation)  
 (preparation of)  
 RN 855242-80-7 HCAPLUS  
 CN 1,1,2,4-Butanetetra-carboxylic acid, 3-methyl- (CA INDEX NAME)



L7 ANSWER 16 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1932:23616 HCAPLUS

DOCUMENT NUMBER: 26:23616

ORIGINAL REFERENCE NO.: 26:2486h-i, 2487a-b

TITLE: Biochemistry of microorganisms. XVI. Production from dextrose by *Penicillium spiculisporem*, Lehman of a new polybasic fatty acid,  $\text{C}_{17}\text{H}_{28}\text{O}_6$  (the lactone of  $\gamma$ -hydroxy- $\beta\delta$ -dicarboxypentadecoic acid)

AUTHOR(S): Clutterbuck, P. W.; Raistrick, H.; Rintoul, M. L.

SOURCE: Trans. Roy. Soc. (London) (1931), B220, 301-30

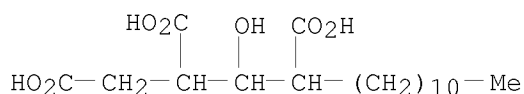
DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB *P. spiculisporem*, Lehman, produces succinic acid and  $\gamma$ -hydroxy- $\beta\delta$ -dicarboxypentadecolactone (I), m.  $145-6^\circ$ ,  $[\alpha]_{5461} -14.76^\circ$  (Na salt,  $[\alpha]_{5461} +14.98^\circ$ ). Hydrolysis with dilute NaOH opens the lactone ring, giving the hydroxytricarboxy acid (II), m.  $134-5^\circ$  [Ac derivative, unstable], reconverted into (I) on heating. (I) or (II) at  $180^\circ$  eliminates 1 or 2H<sub>2</sub>O resp., and gives the dehydrate of (I) (III), m.  $41^\circ$ , hydrolyzed by alkali to an isomeride of (I), a tricarboxylic acid (IV), m.  $87^\circ$ , which reverts to (III) at  $100^\circ$ . With diazomethane (I) yields a Me<sub>2</sub> ester (V); with diazoethane (II) gives an Et<sub>3</sub> ester (VI). (V) with aqueous NH<sub>3</sub> gives a Me ester diamide, m.  $178.5^\circ$ , and Me<sub>2</sub> ester amide, m.  $78.5^\circ$ . Fusion of (I) with KOH gives lauric, oxalic and succinic acids and CO<sub>2</sub>. Oxidation of (I) with acid KMnO<sub>4</sub> or of (II) with KMnO<sub>4</sub> in COMe<sub>2</sub> yields  $\gamma$ -ketopentadecoic acid (VII), m.  $92.6^\circ$ , which by Clemmensen reduction gives pentadecoic acid. Oxidation of NH<sub>4</sub> pentadecoate with H<sub>2</sub>O<sub>2</sub> gave (VII). (VII) was synthesized as follows: n-octyl iodide was condensed with Et malonate to Et n-octylmalonate, b17

169°; n-octylmalonic acid, m. 108°, decomp. 140°, was heated to remove CO<sub>2</sub>, thus forming n-decoic acid, m. 31°. Et n-decoate, b17 131°, was reduced with Na to decyl alcohol, b16 127° and the latter with HI gave decyl iodide, b15 132°. Condensation of the iodide with Et acetoacetate in presence of Na yielded Et α-acetyl-n-dodecoate, b16 170°; the latter with γ-carbomethoxypropionyl chloride and Na gave (VII). (VII) was also isolated from the metabolism solution

IT 872271-86-8, 1,2,4-Pentadecanetricarboxylic acid, 3-hydroxy-  
(and γ-lactone and other derivs. and isomers)  
RN 872271-86-8 HCAPLUS  
CN 1,2,4-Pentadecanetricarboxylic acid, 3-hydroxy- (3CI) (CA INDEX NAME)



L7 ANSWER 17 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1931:16419 HCAPLUS

DOCUMENT NUMBER: 25:16419

ORIGINAL REFERENCE NO.: 25:1802h-i,1803a-i

TITLE: Course of addition of sodium enol alkylmalonic and sodium enol alkylcyanoacetic esters to unsaturated esters

AUTHOR(S): Michael, Arthur; Ross, John

SOURCE: Journal of the American Chemical Society (1931), 53, 1150-72  
CODEN: JACSAT; ISSN: 0002-7863

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 25:16419

AB cf. C. A. 25, 82. the course of the addition of a derivative of the Na enol malonic ester type to an α, β-unsatd. ester proceeds according to the law of chemical neutralization. That addition product is therefore formed in which the positive energy of the Na atom is best neutralized, since the maximum energy degradation is thus realized. In the addition of Na enol alkylmalonic esters to fumaric ester, the alkyl group migrates so that the addendum parts are alkyl and -C(CO<sub>2</sub>Et):C(ONa)OEt; in the addition of Na enol alkylcyanoacetic esters to crotonic ester, the addendum parts are similarly alkyl and -C(CN):C(ONa)OEt. In the addition to crotonic ester, the yield of addition product was greater in the case of Na enol methylcyanoacetic ester (50%) than with Na enol ethylcyanoacetic ester (.33%). However, the yield of addition product is determined by several complex

factors so that it is not possible from these expts. to make any statement regarding the relative ease of migration of the Me and Et groups in these addition reactions. The maximum chemical neutralization of the Na is realized

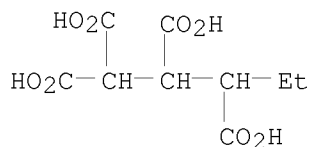
by

the Na remaining attached to the carbonyl O atom in the malonic or cyanoacetic groups. The Na atom, however, becomes better neutralized than it was in the addendum by the spatial negative influence of the acquired CO<sub>2</sub>Et group. Esters of the type CHX (CO<sub>2</sub> Et)CHYCZ(CO<sub>2</sub>Et)<sub>2</sub> (where X = R or H, Y = R, H or CO<sub>2</sub>Et and Z = R or H, also R is an alkyl or aryl radical),

apparently form enolic Na derivs. containing the group  $-\text{CH}:\text{C}(\text{ONa})\text{OEt}$ , but these immediately decompose to form ethylenic  $\alpha,\beta$ -esters and Na alkylmalonic esters. The retrogression of the free ester (A) by Na is probably due to the formation of such an enolic Na derivative. It would follow from this relationship that the Na atom involved in the addition reactions specified above cannot migrate during the addition process. However, if the single  $\text{CO}_2\text{Et}$  group of (A) can form an enolate, an amount of Na corresponding to the relative acidity of the 2 enolic forms of ester (A) ( $Z = \text{H}$ ), would migrate to the single  $\text{CO}_2\text{Et}$  group according to the partition principle, and spontaneous retrogression would lead to the reformation of a further quantity of the Na enol derivative. Thus, by this process, in agreement with the partition principle, an apparent balanced state would be achieved between the 2 possible stable Na derivs. It has not been found possible to obtain addition to the  $\gamma$ -C atom of a  $\beta,\gamma$ -unsatd. ethylenic ester or nitrile. Addition occurs in the case of allyl cyanide and styrylacetic ester at the  $\alpha$ - and  $\beta$ -C atoms with migration of an  $\alpha$ -H atom.  $\text{CH}_2(\text{CO}_2\text{Et})_2$  and  $(\text{CHCO}_2\text{Et})_2$  with a little  $\text{EtONa}$  give 90% of the Et ester, b5  $187^\circ$ , of propane- $\alpha,\beta,\gamma,\gamma$ -tetracarboxylic acid, m.  $153^\circ$ . The ester (22 g.) in an  $\text{Et}_2\text{O}$  suspension of anhydrous  $\text{EtONa}$ , methylated with 10 g.  $\text{MeI}$ , gives 21 g. of the Et ester, ha  $180^\circ$ , of butane- $\alpha,\beta,\gamma,\gamma$ -tetracarboxylic acid, m.  $170^\circ$ . Pentane- $\beta,\gamma,\delta,\delta$ -tetracarboxylic acid, m.  $166^\circ$ , results from the saponification of the ester obtained from the butane derivative and  $\text{MeI}$  with  $\text{EtONa}$  at  $0^\circ$ . The propane derivative and  $\text{EtI}$  with  $\text{EtONa}$  give the Et derivative, b3  $180^\circ$ , of pentane- $\alpha,\beta,\gamma,\gamma$ -tetracarboxylic acid, m.  $177^\circ$ . Pentane- $\alpha,\alpha\beta,\gamma$ -tetracarboxylic acid m.  $179^\circ$  (decomposition). Hexane- $\beta,\beta,\gamma,\delta$ -tetracarboxylic acid, m.  $170^\circ$ , is formed from the Et ester of the last-named acid,  $\text{MeI}$  and  $\text{EtONa}$  at  $0^\circ$ . Addition of  $\text{MeC}(\text{CN}):\text{C}(\text{ONa})\text{OEt}$  to  $\text{MeCH}:\text{CHCO}_2\text{Et}$  gives 50% of the Et ester, b3  $145-8^\circ$ , of  $\alpha,\beta$ -dimethyl- $\gamma$ -cyanoglutaric acid, which could not be crystallized; the corresponding  $\gamma$ -carboxy derivative m.  $142^\circ$  and is not identical with the acid obtained from tiglic acid.  $\beta,\gamma$ -Dimethyl- $\gamma$ -cyanoglutaric acid m.  $152^\circ$ .  $\text{EtC}(\text{CN}):\text{C}(\text{ONa})\text{OEt}$  and  $\text{MeCH}:-\text{CHCO}_2\text{Et}$  give 33% of the Et ester, b3  $153^\circ$ , of  $\gamma$ -cyano- $\alpha$ -ethyl- $\beta$ -methylglutaric acid, m.  $147^\circ$ ; this gives with  $\text{HCl}$  a mixture of  $\alpha$ -ethyl- $\beta$ -methylglutarimides, m.  $92^\circ$  and  $102^\circ$ ; hydrolysis of the imide, m.  $92^\circ$ , gives cis- $\alpha$ -ethyl- $\beta$ -methylglutaric acid, m.  $88^\circ$ . Complete hydrolysis of the above ester gives  $\alpha$ -ethyl- $\beta$ -methyl- $\gamma$ -carboxyglutaric acid, m.  $147^\circ$ .  $\text{MeC}:\text{CEtCO}_2\text{H}$  and  $\text{NCCH}:\text{C}(\text{ONa})\text{OEt}$  give 60% of the Et ester, b4  $152^\circ$ , of  $\alpha$ -ethyl- $\beta$ -methyl- $\gamma$ -cyanoglutaric acid, m.  $132^\circ$ ; complete hydrolysis gives the  $\gamma$ -carboxy derivative, m.  $141^\circ$ . Ethylation of  $\gamma$ -cyano- $\beta$ -methylglutaric ester gives the Et ester, b4  $152^\circ$ , of  $\gamma$ -ethyl- $\beta$ -methyl- $\gamma$ -cyanoglutaric acid, m.  $139^\circ$ ;  $\gamma$ -ethyl- $\beta$ -methyl- $\gamma$ -carboxyglutaric acid could not be crystallized.  $\text{CH}_2:\text{CHCH}_2\text{CN}$  and  $\text{NCCH}:\text{C}(\text{ONa})\text{OEt}$  give 90% of  $\beta$ -methyl- $\gamma$ -carbethoxyglutaronitrile, b3  $160^\circ$ ;  $\text{MeC}(\text{CN}):\text{C}(\text{ONa})\text{OEt}$  gives  $\alpha,\beta$ -dimethyl- $\gamma$ -carbethoxyglutaronitrile, b3  $152^\circ$ .  $\text{PhCH}:\text{CHCH}_2\text{CO}_2\text{Et}$  and  $\text{CNCH}_2-\text{CO}_2\text{Et}$  with  $\text{EtONa}$  give Et  $\beta$ -benzyl- $\gamma$ -cyanoglutarate, b3  $193^\circ$ ; hydrolysis gives  $\beta$ -benzyl- $\gamma$ -carboxyglutaric acid, m.  $158^\circ$ ; the Na derivative of the ester and  $\text{MeI}$  give Et

$\gamma$ -methyl- $\beta$ -benzyl- $\gamma$ -cyanoglutarate, b<sub>3</sub> 194°; alkaline hydrolysis gives  $\gamma$ -methyl- $\beta$ -benzyl- $\gamma$ -carboxyglutaric acid, m. 177° (decomposition), giving  $\alpha$ -methyl- $\beta$ -benzylglutaric acid, m. 139°. PhCH:CHCH<sub>2</sub>CO<sub>2</sub>Et and MeCH(CO<sub>2</sub>Et)<sub>2</sub> with EtONa give the Et ester, b<sub>3</sub> 197° of  $\alpha$ -methyl- $\beta$ -benzyl- $\gamma$ -carboxyglutaric acid, which exists in 2 forms, m. 197° and 118°; heating above their m. p. gives 2  $\alpha$ -methyl- $\beta$ -benzylglutaric acids, m. 139° (cf. above) and 97°. PhMeCHCHO and CH<sub>2</sub>(CO<sub>2</sub>H)<sub>2</sub> in EtOH give a mixture of the Et esters, b<sub>10</sub> 156° and b<sub>10</sub> 175-82°, of  $\gamma$ -methyl- $\gamma$ -phenylbutenoic acid (I), did not crystallize, and  $\gamma$ -methyl- $\gamma$ -phenyl- $\alpha$ -carboxybutenoic acid, m. 151°. I and NCCH<sub>2</sub>CO<sub>2</sub>Et with EtONa give Et  $\gamma$ -methyl- $\gamma$ -phenyl- $\alpha$ -cyano- $\beta$ -aceticbutyrate, b<sub>3</sub> 198°; alkaline hydrolysis gives  $\gamma$ -methyl- $\gamma$ -phenyl- $\alpha$ -carboxy- $\beta$ -aceticbutyric acid, m. 162° (decomposition), giving  $\gamma$ -methyl- $\gamma$ -phenyl- $\beta$ -aceticbutyric acid, m. 88°.

IT 858834-84-1P, 1,1,2,3-Propanetetracarboxylic acid, 3-ethyl-  
 RL: PREP (Preparation)  
 (preparation of)  
 RN 858834-84-1 HCAPLUS  
 CN INDEX NAME NOT YET ASSIGNED



L7 ANSWER 18 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1928:37608 HCAPLUS

DOCUMENT NUMBER: 22:37608

ORIGINAL REFERENCE NO.: 22:4474g-i,4475a-g

TITLE: Decomposition of the six-carbon chain of adipic acid.  
 II

AUTHOR(S): v. Braun, Julius; Jostes, Fritz; Wagner, Hans

CORPORATE SOURCE: Univ. Frankfurt a. M.

SOURCE: Berichte der Deutschen Chemischen Gesellschaft  
 [Abteilung] B: Abhandlungen (1928), 61B,  
 1423-31

CODEN: BDCBAD; ISSN: 0365-9488

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB cf. C. A. 21, 60; Fuson, C. A. 22, 2144. F. reports that NH<sub>2</sub>Et and its homologs with (CH<sub>2</sub>CHBrCO<sub>2</sub>Et)<sub>2</sub> (I) gives, along with AcCO<sub>2</sub>Et, Et<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>Et (II) and not MeCH(NEt<sub>2</sub>)CO<sub>2</sub>Et (III), as v. B. and his co-workers believed. Their belief was based on the fact that the alc. (IV) formed by reduction of the substance was different from the Et<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OH (V), which had been obtained 12 yrs. before from Et<sub>2</sub>NH and BrCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OBz and subsequent saponification of the resulting Et<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OBz; the IV and V boiled at very nearly the same temperature, to be sure, but IV gave an only slightly hygroscopic methiodide, m. 188°, while V yielded a

quite hygroscopic methiodide, m. 174°. Having occasion recently to prepare some of the latter methiodide, the authors found that the old preparation

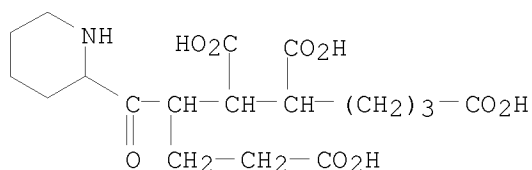
must still have been somewhat impure, for 2 more crystns. raised the m. p. to 188° and the hygroscopicity diminished somewhat. Again, III gives a methiodide which, contrary to Fuson, is not oily but m. 70° and depresses the m. p. (80°) of the methiodide of the ester (II) obtained from I about 50°, and the methiodide, m. 263°, of  $\text{MeCH}(\text{NEt}_2)\text{CH}_2\text{OH}$  (obtained from III) materially lowers the m. p. of the methiodide m. 188° obtained in either of the 2 ways described above. The authors now therefore agree completely with Fuson as to the course of the decomposition of I, but while F. believes that the determining factor

is the tendency to the formation of a 4-C ring, the authors still think that it is the form of the amine reacting with the I. This conclusion was based on a comparison of the behavior of  $\text{Me}_2\text{NH}$  and piperidine on the one hand and of  $\text{Et}_2\text{NH}$  and copellidine on the other; the more disk-like form of the first 2 bases allows them to substitute both Br atoms (spatially near to each other) in I, while the branched, more 3-dimensional form of the last 2 bases either prevents or represses such a substitution; if one Br atom is substituted, there is started a transition reaction which results either in the formation of a 4-membered ring, which decomps. further, or of a double bond, which persists. The formation of a double bond with  $\beta$ -methyladipic acid had already been shown to be very probable and a very careful study of I has now shown that a very small quantity of the compound  $\text{EtO}_2\text{CCH}(\text{NEt}_2)\text{CH}_2\text{CH}:\text{CHCO}_2\text{Et}$  is formed in this case too. A study was then made of bases which may be considered as intermediate between those given above. Below are given the number of mols. of basic propionic ester per 100 mols. of diaminoadipic ester formed by I with various amines:  $\text{Me}_2\text{NH}$  0,  $\text{MeNHEt}$  10,  $\text{MeNHPr}$  20,  $\text{MeNHCHMe}_2$  200,  $\text{Et}_2\text{NH}$  1600,  $(\text{RCH}_2\text{CH}_2)_2\text{NH}$  > 1600, piperidine 0,  $\beta$ -methylpiperidine 200,  $\alpha$ -methylpiperidine 570, decahydroquinoline 520,  $\alpha$ -methyl- $\beta'$ -ethylpiperidine 500. These values show clearly that it is the progressive alkylation of the  $\alpha$ -C atoms to the N and not of the more distant atoms which is of the greatest influence on the tendency of the bases to produce decomposition III, b13 69-71°, gives with Na and alc. 40% of the alc., b13 56-8°. Me  $\beta$ -[methylethylamino]propionate, b13 75-80°, and impure di-Et  $\alpha, \alpha'$ -bis[methylethylamino]adipate, b13 160-5° (found C 59.95, H 9.50%), are obtained in the ratio 1:20 from I and  $\text{MeNHEt}$ .  $\text{MeNHPr}$ , b. 61-2°, is obtained almost quant. from  $\text{PhSO}_2\text{NHMe}$  and  $\text{PrI}$  and subsequent hydrolysis with concentrated  $\text{HCl}$  at 160° of the resulting  $\text{PhSO}_2\text{NMePr}$ , b13 182-3°, which is obtained in 80% yield;  $\text{HCl}$  salt, hygroscopic, m. 150°; phenylurea, m. 95°. With I it gives 5 parts Et  $\beta$ -[methylpropylamino]propionate, b13 83-5° ( $\text{HCl}$  salt, hygroscopic, m. 111-2°; picrate, m. 75-7°), and 36 parts of the impure diaminoadipate. N-Isopropylbenzenesulfonamide, b13 190°; methylisopropylbenzenesulfonamide, b13 175° (yield, 85%). Methylisopropylamine, b. 50°, d419 0.7026; picrate, m. 135°; chloroplatinate, m. 185-9°;  $\text{HCl}$  salt, hygroscopic, m. 77°; Ac derivative, b13 69-70°; Bz derivative, b12 144°; phenylurea, m. 131°; phenylthiourea, m. 120°. With I it yields about equal parts of Et  $\beta$ -[methylisopropylamino]propionate, b13, 84-6° (picrate, m. 85-6°), and impure diaminoadipate.  $\beta$ -Pipicoline, b. 125° (Bz derivative, m. 44-5°;  $\text{CdI}_2$  compound, m. 144°), gives with I about equal parts of Et  $\beta$ - $\beta'$ -pipicolylpropionate,



b13 111-2° (HCl salt, m. 167-9°; picrate, m. 98-9°), and di-Et  $\alpha, \alpha'$ -di- $\beta''$ -pipecolyladipate, m. 61-3°, mol. weight in camphor 388 (HCl salt, m. 191°; picrate, m. 196°).  $\alpha$ -Pipicoline gives relatively more (about 75% of the total product) of the Et  $\beta$ - $\alpha'$ -pipecolylpropionate, b14 117-9°; picrate, m. 123°. trans-Decahydroquinoline gives about 3 parts Et  $\beta$ -[decahydroquinolyl]propionate, faintly yellow, b13 155-6° (HCl salt, m. 165-7°; picrate, m. 102°), and 25% pure di-Et  $\alpha, \alpha'$ -di[decahydroquinolyl]adipate, b13 about 220°, m. 107-8°.

IT 860582-64-5, Adipic acid,  $\alpha, \alpha'$ -di- $\beta''$ -pipecolyl-  
(diethyl ester and its derivs.)  
RN 860582-64-5 HCAPLUS  
CN 1,4,5,8-Octanetetra-carboxylic acid, 3-(2-piperidinylcarbonyl)- (CA INDEX NAME)



L7 ANSWER 19 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1924:8308 HCAPLUS

DOCUMENT NUMBER: 18:8308

ORIGINAL REFERENCE NO.: 18:1128e-i

TITLE: Course of the reduction of pyridinecarboxylic acids to nitrogen-free products

AUTHOR(S): Mumm, Otto; Brodersen, Karl

SOURCE: Berichte der Deutschen Chemischen Gesellschaft [Abteilung] B: Abhandlungen (1923), 56B, 2295-301

CODEN: BDCBAD; ISSN: 0365-9488

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

GI For diagram(s), see printed CA Issue.

AB From 10 g. 2-methyl-6-phenylcinchomeronic acid (I) in boiling NaOH slowly treated in a current of CO<sub>2</sub> with 75 g. of 2.5% Na-Hg (2 mols.) is obtained, together with 2.5g. unchanged I, 2.5 g.  $\alpha$ -acetyl- $\beta$ -benzoylpropionic acid ( $\beta$ -acetyl- $\beta'$ -benzoylisobutyric acid) (II), BzCH<sub>2</sub>CH(CH<sub>2</sub>Ac)CO<sub>2</sub>H, m. 83°, solidifies and m. again 135° (m. p. of the lactone, which begins to be formed even at room temperature in the air); phenylhydrazone, m. 204°. II can also be synthesized from BzCH<sub>2</sub>CO<sub>2</sub>Et and AcCH:CHCO<sub>2</sub>Et through the compound BzCH(CO<sub>2</sub>Et)CH(CH<sub>2</sub>Ac)CO<sub>2</sub>Et. With 4 mols. Na-Hg, 5 g. I gives 3.2 g.  $\alpha$ -acetyl- $\gamma$ -phenylbutyrolactone (III) or  $\alpha$ -benzoylmethyl- $\gamma$ -valerolactone (IV), exceedingly viscous oil, b0.5-0.6 198-220°. With 8 mols. Na-Hg is obtained  $\alpha$ -[ $\beta'$ -hydroxypropyl]- $\gamma$ -phenyl- $\gamma$ -butyrolactone (V), yellowish, semi-solid, glassy mass, gives on long boiling with Ba(OH)<sub>2</sub> the Ba salt, (C<sub>18</sub>H<sub>17</sub>O<sub>4</sub>)<sub>2</sub>Ba, of the acid as a glassy brittle mass which with AgNO<sub>3</sub> yields the Ag salt, flocculent precipitate, turns brown in the light, m. 185° (decomposition); the lactone, treated 3 hrs.

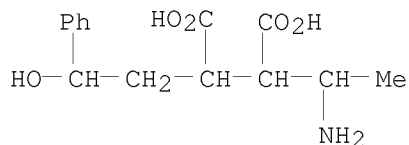
in absolute alc. on the H<sub>2</sub>O bath with dry HCl and allowed to stand 36 hrs. isomerizes into  $\alpha$ -[ $\beta'$ -hydroxy- $\beta'$ -phenylethyl]- $\gamma$ -valerolactone (VI), faintly yellowish oil, b<sub>13</sub> 180-3°, yields the same Ag salt as the isomer V. On fractionating twice under 0.6 mm. V undergoes a peculiar degradation of its side chain, 5 g. yielding 2.8 g.  $\alpha$ -methyl- $\gamma$ -phenylbutyrolactone, b<sub>0.6</sub> 180-210°, m. about 60°, forms a Ba salt (C<sub>11</sub>H<sub>13</sub>O<sub>3</sub>)<sub>2</sub>Ba, m. 236-50° (decomposition), and a Ag salt. With 16 mols. Na-Hg I in ice-cold NaOH yields a small amount of 1-phenyl-1-hydroxy-5-amino-hexane-3,4-dicarboxylic acid (VII), m. 260° (which easily loses 1 mol. H<sub>2</sub>O and then forms a HCl salt, C<sub>14</sub>H<sub>18</sub>O<sub>4</sub>NC<sub>1</sub>, m. 220°), and of an isomer, extremely hygroscopic powder. The above results can hardly leave any doubt that the first step in the reduction of I is the formation of the dihydro derivative PhC:CH.CH(CO<sub>2</sub>H).C(CO<sub>2</sub>H):CMe.NH, which with NaOH gives PhCOCH<sub>2</sub>CH(CO<sub>2</sub>H)C(CO<sub>2</sub>H):CMeNH<sub>2</sub> or PhC(NH<sub>2</sub>):CHCH(CO<sub>2</sub>H)CH(CO<sub>2</sub>H)COMe (or probably both) and these in turn yield PhCOCH<sub>2</sub>CH(CO<sub>2</sub>H)CH(CO<sub>2</sub>H)COMe with NaOH and VII and its isomer with H.

IT 861323-17-3P, 3,4-Hexanedicarboxylic acid, 5-amino-1-hydroxy-1-phenyl-

RL: PREP (Preparation)  
(preparation of)

RN 861323-17-3 HCAPLUS

CN Butanedioic acid, 2-(1-aminoethyl)-3-(2-hydroxy-2-phenylethyl)- (CA INDEX NAME)



L7 ANSWER 20 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1922:24645 HCAPLUS

DOCUMENT NUMBER: 16:24645

ORIGINAL REFERENCE NO.: 16:4187d-i, 4188a-b

TITLE: Ring-chain tautomerism. III. The occurrence of tautomerism of the three-carbon (glutaconic) type between a homocyclic compound and its unsaturated open-chain isomeride

AUTHOR(S): Ingold, Christopher Kelk; Perren, Edward Arthur; Thorpe, J. F.

SOURCE: Journal of the Chemical Society, Transactions (1922), 121, 1765-89  
CODEN: JCHTA3; ISSN: 0368-1645

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB cf. C. A. 16, 912, 2141, 3065. A general discussion of the conditions which govern the occurrence of any Michael reaction: The acetic ester must contain a neg. substituent such as a -CO<sub>2</sub>Et or -CN group to confer the necessary mobility on the adjacent H atom. The acrylic ester involved should be only lightly substituted. The presence of a  $\beta$ -substituent, particularly 2  $\beta$ -substituents, considerably reduces the tendency towards condensation. The effect is apparently a spatial one. The

presence of an  $\alpha$ -substituent greatly inhibits condensation, the magnitude of the effect depending on the size of the group. The simple spatial relationships noted above break down in the case of strongly electroneg. substituents, such as the  $\text{-CO}_2\text{Et}$  and  $\text{-CN}$  groups, which inhibit condensation very slightly. Et  $\alpha$ -cyano- $\gamma$ -ethylglutaconate (from Et  $\alpha$ -formylbutyrate, b15  $100^\circ$ , and  $\text{CHNa}(\text{CN})\text{CO}_2\text{Et}$ ), b14  $163^\circ$ . The  $\gamma$ -Ph derivative, b14  $200-5^\circ$ . The self-condensation of glutaconic esters alone and with piperidine was tested for a number of compds. The following esters appeared not to have given any condensation product after a year:  $\text{EtO}_2\text{CCH}_2\text{CH:CHCO}_2\text{Et}$ ,  $\text{EtO}_2\text{CCH}_2\text{C}(\text{CO}_2\text{Et})\text{:CHCO}_2\text{Et}$ ,  $\text{EtO}_2\text{CCHMeCH:CHCO}_2\text{Et}$ ,  $\text{EtO}_2\text{CCH}_2\text{CMe:CHCO}_2\text{Et}$ ,  $\text{EtO}_2\text{CCH}(\text{CN})\text{CMe:CHCO}_2\text{Et}$ ,  $\text{EtO}_2\text{CCH}(\text{CN})\text{CMe:CEtCO}_2\text{Et}$ ,  $\text{EtO}_2\text{CCH}(\text{CN})\text{CH:CMeCO}_2\text{Et}$ ,  $(\text{EtO}_2\text{C})_2\text{CHCH:CEtCO}_2\text{Et}$ ,  $\text{EtO}_2\text{CCH}(\text{CN})\text{CH:CEtCO}_2\text{Et}$  and  $\text{EtO}_2\text{CCH}(\text{CN})\text{CH:CPhCO}_2\text{Et}$ . The self-condensation of  $(\text{EtO}_2\text{C})_2\text{CHCH:CHCO}_2\text{Et}$  is practically complete in a week in the presence of a catalyst, giving Et 2,2,4,4,-tetracarboxycyclobutane-1,3-diacetate (cf. Guthzeit, C. A. 4, 906). In the condensation of  $(\text{EtO}_2\text{C})_2\text{CHCH:C}(\text{CO}_2\text{Et})_2$  in the presence of piperidine, Et piperidinomethylenemalonate also is formed, pale yellow rhombohedral plates, m.  $216^\circ$  (decomposition). Et 2,2,4,4-tetracarboxycyclobutane-1,3-dimalonate, m.  $103^\circ$ , upon fusing, or maintaining in solution with piperidine for a long period, gives an equilibrium mixture, containing about

80% of

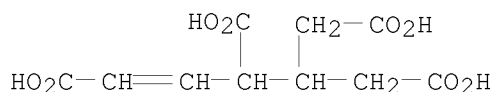
the cyclobutane ester and about 10% of Et  $\alpha,\alpha,\gamma,\gamma,\epsilon,\epsilon$ -hexacarboxy- $\Delta\alpha$ -pentene- $\delta$ -malonate, the constitution of which was established by hydrolysis to  $\alpha,\gamma,\epsilon$ -tricarboxy- $\Delta\alpha$ -pentene- $\delta$ -acetic acid, sirupy, which, on oxidation, gave  $\text{CH}(\text{CH}_2\text{CO}_2\text{H})_3$ . Et 2,4-dicyano-2,4-dicarboxycyclobutane-1,3-di- $\alpha$ -propionate, from the condensation of  $\text{EtO}_2\text{CCH}(\text{CN})\text{CH:CMeCO}_2\text{Et}$ , long silky needles and small glistening plates, both m.  $87^\circ$ . With 20% HCl this gives a mixture of trans-2,4-dicarboxycyclobutane-1,3-di- $\alpha$ -propionic acid, m.  $251^\circ$ , separated from the cis-acid by treatment with  $\text{AcCl}$ , which converted the latter into its anhydride; cis-acid, m.  $144-5^\circ$ . Condensation of different esters gave: Et 2-cyano-2,4,4-tricarboxycyclobutane-1-malonate-3- $\alpha$ -propionate, viscous liquid, b15  $260^\circ$ . Et 2,2,4,4-tetracarboxycyclobutane-1-malonate-3-acetate, long needles, m.  $92^\circ$ . Et 2-cyano-2,4,4-tricarboxycyclobutane-1-acetate-3- $\alpha$ -propionate, stout prisms, m.  $81^\circ$ . 2,4-Dicarboxycyclobutane-1,3-diacetic anhydride, by boiling either the  $\alpha$ - or  $\beta$ -form of the acid with  $\text{AcCl}$ , m.  $235^\circ$ . On heating the  $\beta$ -acid with 30% HCl at  $200^\circ$  for 5 h., it is converted to the  $\epsilon$ -acid, m.  $223^\circ$ .

IT 861342-50-9P,  $\Delta 1$ -1,3,5-Pentenetricarboxylic acid, 4-(carboxymethyl)-

RL: PREP (Preparation)  
(preparation of)

RN 861342-50-9 HCAPLUS

CN 1-Pentene-1,3,5-tricarboxylic acid, 4-(carboxymethyl)- (CA INDEX NAME)



L7 ANSWER 21 OF 22 HCAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1922:20408 HCAPLUS

DOCUMENT NUMBER: 16:20408

ORIGINAL REFERENCE NO.: 16:3467c-g

TITLE: Synthesis of the polyacetic acids of methane. VII.  
Isobutylene- $\alpha,\gamma,\gamma'$ -tricarboxylic acid and methanetetraacetic acid

AUTHOR(S): Ingold, C. K.; Nickolls, L. C.

SOURCE: Journal of the Chemical Society, Transactions (1922), 121, 1638-48  
CODEN: JCHTA3; ISSN: 0368-1645

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB cf. C. A. 16, 3070. Directions are given for the preparation of  $\text{CO}(\text{CH}_2\text{CO}_2\text{H})_2$  and its Et ester. Ethyl  $\beta$ -chloroglutaconate, mobile oil, with sweet fruity odor, b11, 136-7°. With  $\text{CHNa}(\text{CO}_2\text{Et})_2$  this condenses to ethyl isobutylene- $\alpha,\gamma,\gamma,\gamma'$ -tetracarboxylate (A), b12 220-2°, which, on hydrolysis with 20% HCl for 48 hrs., gave isobutylene- $\alpha,\gamma,\gamma'$ -tricarboxylic acid,  $(\text{HO}_2\text{CCH}_2)_2\text{C}:\text{CHCO}_2\text{H}$ , clusters of needles, m. 140°. Ethyl ester, mobile oil, b11 174-5°. The action of  $\text{NH}_3$  on the acid gave 2,6-dihydroxypyridine-4-acetamide (imide-amide of the acid), needles, m. 228°; air must be carefully excluded during the reaction because of oxidation to a blue-green compound. The constitution of the acid was established by reduction with Na-Hg to  $\text{CH}-(\text{CH}_2\text{CO}_2\text{H})_3$ . Ethyl w-cyanomethanetetraacetate, obtained by condensing the Et ester from the crude hydrolysis product of A with  $\text{CH}_2(\text{CN})\text{CO}_2\text{Et}$ , pale yellow, viscous oil, b13 234-5°. When the ester was allowed to stand with an equal volume of cold concentrated  $\text{H}_2\text{SO}_4$ , for 12 hrs., then diluted with 2 vols.  $\text{H}_2\text{O}$  and heated

for 7 hrs., and finally treated with  $\text{HNO}_2$  to remove the last traces of N, it gave methanetetraacetic acid, stout glistening octahedrons, m. 248°. Barium salt, long felted needles, which are very slightly soluble. Silver salt, curdy precipitate, unusually slightly soluble, and is stable

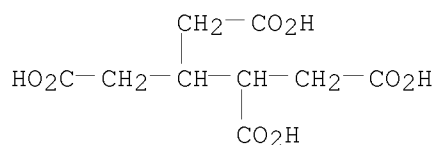
towards light. Dianhydride, by heating with  $\text{AcCl}$  in a sealed tube for 6 hrs., hexagonal plates, m. 284°. Ethyl  $\beta$ -cyanoisopentane- $\alpha,\beta,\gamma,\gamma'$ -tetracarboxylate by the action of the Na derivative of  $\text{NCC}-(\text{CO}_2\text{Et})_3$  with  $\text{ICH}_2\text{CO}_2\text{Et}$ , viscous oil, b10 222-4°. Isopentane- $\alpha,\beta,\delta,\delta'$ -tetracarboxylic acid, needles, m. 182°.

IT 861328-06-5P, Isopentane-,  $\alpha,\beta,\delta,\delta'$ -tetracarboxylic acid

RL: PREP (Preparation)  
(preparation of)

RN 861328-06-5 HCAPLUS

CN 1,2,4-Butanetricarboxylic acid, 3-(carboxymethyl)- (CA INDEX NAME)



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ACCESSION NUMBER: 1912:24399 HCAPLUS

DOCUMENT NUMBER: 6:24399

ORIGINAL REFERENCE NO.: 6:3410c-i

TITLE: The Condensation of Ethyl Sodiomalonate with Ethyl Citraconate and the Synthesis of  $\beta$ -Methyltricarballic Acid

AUTHOR(S): Hope, Edward

CORPORATE SOURCE: Manchester

SOURCE: Proc. Chem. Soc. (1912), 28, 93

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB The condensation of  $\text{CHNa}(\text{CO}_2\text{Et})_2$  (a), and  $\text{EtO}_2\text{CCH} : \text{CMeCO}_2\text{Et}$  (b), gives rise to cyclopentane derivs., not cyclobutanes (cf. Michael, J. prakt. Chemical, [2] 35, 3 54; 43, 395; 45, 57; 49, 20; Ber., 33, 3756; 36, 576). In  $\text{Et}_2\text{O}$  or  $\text{C}_6\text{H}_6$ , Et butane- $\alpha, \beta, \delta$ -tetracarboxylate,  $\text{CH}(\text{CO}_2\text{Et})_2\text{CH}(\text{CO}_2\text{Et})\text{CHMeCO}_2\text{Et}$  (c), is formed; viscid oil, b14 198°, not  $\text{CH}(\text{CO}_2\text{Et})_2\text{CMe}(\text{CO}_2\text{Et})\text{CH}_2\text{CO}_2\text{Et}$ . With alc. KOH, butane- $\alpha, \alpha, \beta, \delta$ -tetracarboxylic acid, m. 154-6° (decompose) is formed. On hydrolysis with concentrate HCl, the isomeric  $\alpha$ -methyltricarballic acids were obtained. Et  $\alpha$ -methyltricarballylate,  $\text{EtO}_2\text{CCHMeCH}(\text{CO}_2\text{Et})\text{CH}_2\text{CO}_2\text{Et}$ , b24 180-1°. (c), Na and MeI gave Et pentane- $\beta, \delta, \delta, \delta$ -tetracarboxylate, b15 196°, also formed from the Na derivative of (d) (below) and MeI, or in small yields from Et itaconate and  $\text{CHNa}(\text{CO}_2\text{Et})_2$ . Hydrolysis with HCl gave a mixture of acids from which  $\alpha, \alpha'$ -dimethyltricarballic acid was isolated. In EtOH, at room temperature, (a) and (b) give chiefly Et butane- $\alpha, \beta, \delta, \delta$ -tetracarboxylate (d), b13 207-9°, which was also prepared from (a) and Et itaconate,  $\text{CH}_2 : \text{C}(\text{CO}_2\text{Et})\text{CH}_2\text{CO}_2\text{Et}$ . Butane- $\alpha, \beta, \delta$ -tricarboxylic acid, m. 122°, from (d) and HCl. In b. EtOH, (a) and (b) give chiefly 1-methylcyclopentane-2-one-1,3,4-tricarboxylate, (e), identical with Michael's so-called Et dimethylcyclobutanonetricarboxylate and Svoboda's Et 1-methylcyclopentane-3-one-1,5,5-tricarboxylate (Monatsh., 23, 846). It b11 195-200° with some decompose. On hydrolysis with 8% aqueous  $\text{H}_2\text{SO}_4$ , it gives cyclopentanone-4-carboxylic acid. Et itaconate and (a) yield the same products as (a) and (b). (e) may also be prepared from (d) and NaOEt. Ring closure is explained by the assumption that the  $\delta$ -Na derivative of (d) is gradually converted into the  $\alpha$ -Na derivative, which then loses NaOEt. (b) and 0.04 mol. NaOEt gave Et ethoxymethylsuccinate, b14 132-4°. Hydrolysis gave the acid, m. 81-3°. Et  $\alpha$ -methoxy- $\alpha$ -methylsuccinate, b13 113-6°. Hydrolysis yields the acid, m. 90-2°.  $\text{EtO}_2\text{CCH} : \text{CMeCH}_2\text{CO}_2\text{Et}$ , from  $\text{AcCH}_2\text{CO}_2\text{Et}$  and  $\text{NCCHNaCO}_2\text{Et}$ , was heated with KCN and EtOH, and the resulting compound hydrolyzed with  $\text{H}_2\text{SO}_4$  and then esterified, yielding Et  $\beta$ -methyltricarballylate, b15 163-4°. The acid (f), m. 165-6°, and has properties similar to the  $\alpha$ -derivative. The acid prepared by Michael and Auwers (Ber., 24, 2893) was probably the impure  $\alpha$ -acid. (f), heated at low pressure, gives the anhydro acid, b15 220-3, prisms, m. 139-40°, which, with  $\alpha$ - $\text{C}_{10}\text{H}_7\text{NH}_2$ , gives a compound,  $\text{C}_{17}\text{H}_{15}\text{O}_4\text{N}$ , m. 98-100°. (f) may also be prepared direct from  $\text{AcCH}_2\text{CO}_2\text{Et}$ ,  $\text{NCCH}_2\text{CO}_2\text{Et}$ , and 2 mols. KCN in b. EtOH (12 h.). If only 1 mol. KCN is used, Et  $\beta, \gamma$ -dicyano- $\beta$ -methylpropane- $\alpha, \gamma$ -dicarboxylate, b20 200-5°, is formed.

10/564,340

IT 847934-42-3P, 1,1,2,3-Butanetetracarboxylic acid  
RL: PREP (Preparation)  
(preparation of)  
RN 847934-42-3 HCAPLUS  
CN 1,1,2,3-Butanetetracarboxylic acid (CA INDEX NAME)

